

INVESTIGATION OF PROBLEMS ASSOCIATED WITH
SOLID ENCAPSULATION OF HIGH VOLTAGE ELECTRONIC
ASSEMBLIES; ALSO REYNOLDS CONNECTOR STUDY

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FOREWORD

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INVESTIGATION OF PROBLEMS ASSOCIATED WITH SOLID
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ALSO REYNOLDS CONNECTOR STUDY

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ABSTRACT

~~Three~~ interrelated studies involving electric-breakdown-prevention in vacuum and encapsulation of high voltage electronic circuits were undertaken:

- I. Adhesive strengths were measured according to the ASTM D-1002 lap shear method on the Instron Tensile Testing TTM machine. Adhesives were Shell Epon 828 Epoxy, Thiokol Solithane 113, Dow Corning Sylgard 184. Adherents were Solder on Beryllium Copper, Glass Epoxy circuit board, porcelain, and Ferrite. Different types of conservative surface preparations suitable for electronics could improve the lap shear adhesive strength by about a factor of three.

- II. The measurement of the permeation constants of air at ambient room temperature through four different space grade encapsulants was brought to completion. The measured constants permit order of magnitude calculations of the time for pressures in air bubbles to drop to the corona region due to permeation of air through the encapsulants into the vacuum of space. It takes hundreds of years for Emerson and Cuming Stycast 3050 Epoxy, tens of years for Epon 828 Epoxy, weeks through Thiokol Solithane, a few days through Dow Corning Sylgard.

III.

Reynolds Corporation, series 600, high voltage connectors with "L" type cable attached were tested in a vacuum system at various pressures. There is no black or white answer to this system. Yes, the Reynolds 600 high voltage connector-Type "L" cable-system definitely suppresses catastrophic break-down when filled with and surrounded by gas in the corona region of pressures. No, the connector-cable system is not completely noise free. When filled with and surrounded by gas between 2 to 10 torr (the corona region of pressures for this system) there are a few noise counts, about 10 counts/5 minute interval, at a sensitivity of 3×10^{-14} coulombs and up, at 3000 volts, or about the same count rate at sensitivity of 8×10^{-15} coulombs and up, at 2500 volts. There are even some noise counts at atmospheric pressure if the connector-cable system has never been outgased. The prospective user will have to peruse the detailed test results and then ask himself whether his experiment at his sensitivity can tolerate the noise count or whether he can filter it out, or suppress it by lowering his sensitivity.

CHAPTER I ADHESIVE STUDY

Introduction

When an electronic assembly is potted or encapsulated, it is important that adhesion between the potting compound and the electronic components be good. Otherwise, the potting compound will pull away from the components and the newly formed narrow gap will quickly fill with outgasing vapors that might well increase rapidly to pressures of 0.1 torr to 10 torr. In this region, the corona region of pressures, electric breakdown is extremely easy even for very modest potential differences such as 400V, with resulting power loss and electrically noisy circuits. Poor adhesion must therefore be avoided.

When the choice of adhesive and substrates is fixed by electrical considerations, the only variable left to the experimenter is method of surface preparation. This is important because if an adhesive bond is to be formed by the molecular attraction of the adhesive molecules for the substrate molecules then the surface has to be clean. A really, truly clean, fresh surface can only be prepared in ultra high vacuum.¹ But chemical and mechanical cleaning in air can go far toward removal of grease, oxide and scale, so that the adhesive wets the surface.²

The smaller the contact angle, and the greater the wetting area of a drop of the liquid adhesive, the greater the subsequent shear strength of the cured adhesive bond.³ In fact, one

can get an indication of the bondability of a surface immediately after the surface has been cleaned and treated by measuring the contact angle of a drop of water on it.⁴ The most reliable method, however, is to carry out the bonding and then tear it apart with a tensile tester by either the peel or tensile shear or lap shear tests.⁴ Some authors state that the peel test is the best method for determining the quality of a bonding surface.⁵ One study was encountered where tensile-shear testing was used.^{6,7} However, the more usual data encountered in the literature is the lap-shear strength. This is perhaps due to the fact that apparatus for lap-shear specimen preparation and pulling apart are more easily available than for peel testing. In the case of the experiment described in this report the lap-shear testing ASTM D-1002 or a variation thereof was decided upon.

Method

Four substrates were used: Solder on beryllium copper, glass epoxy board, unglazed porcelain, ferrite. The solder on beryllium copper and glass epoxy board was available in 0.063 "thick x 1" wide x 4" long bars. After surface preparation these were bonded with a 0.5" overlap in an alignment jig as shown in Figure 1. The bondline thicknesses were assured by placing two 0.5" long pieces of nichrome steel wire on the faying surface, length of wires parallel to length of bar.

The ferrite was not available in dimensions suitable for lap shear specimens and is, anyhow, much too brittle to be

held directly in the tensile tester jaws without breaking. Ferrite samples were bonded as a single strap to epoxy board strips, as shown in Figure 2(d). The porcelain samples used were so called unglazed streak-plates available in dimensions 1-1/4" wide from Fisher Scientific Co. that had to be sawed in half widthwise to fit into the tensile tester grips, and then lap shear specimens could be prepared in the usual manner. The sawing had to be done on a diamond saw with heavy cutting grease unfortunately. This was extremely hard to remove from the porous porcelain surface afterward, which makes the data of Solithane 113 on porcelain somewhat questionable.

Attempts were made to use the porcelain as the inner layer in various arrangements of a triple lap joint as shown in Figure 2(b) and suggested by Cagle.⁸ This did not work, however, because when a lap shear joint is pulled apart, the sandwich thickness dimension, l , acts as a lever arm for force F , giving rise to a peel torque about points A or B in Figure 2(c). Therefore, the thicker the glue line or the thicker the sandwich the more the peel torque becomes effective. The sandwiches always seemed to come apart at the outer adherent, for values much less than their previously by measured shear strengths, due to the introduction of this peel torque.

The adhesives used were three of the four different space-grade encapsulants, enumerated in Table I below:

The cure schedules were as follows: The Epon 828 was allowed to "set up" at room temperature over night, then cured at 150°F for twelve hours. The Solithane 113 was placed in an

150°F oven immediately after assembly of the lap joints and cured for 15 hours. The Sylgard was allowed to "set up" at room temperature over night, then heated at 150°F for 2 hours.

The adhesive overflow and bond line fillets were always carefully removed with either razor blade or grinding tool, hopefully avoiding damage to the adhesive joint.

TABLE I

Four high voltages "potting" compounds. (Proportions by weight)

EPON EPOXY	Shell, Epon 828 resin, 50% General Mill Versamid 140 hardener, 50% or Miller Stephenson V-40 hardener, 50%	Semi Rigid, Transparent
STYCAST EPOXY	Emerson & Cuming Stycast 3050, 100 parts; Catalyst 9 hardener, 8 parts	Rigid, Opaque
Solithane (Urethane)	Thiokol C113 Resin, 50% Thiokol C113-300 hardener, 50%	Semiflexible, Transparent
SILICONE (Elastomer)	Dow Corning Sylgard 184 or 185 resin, 10 parts; Dow Corning Syl- gard 184 or 185 curing agent, 1 part	Flexible, Transparent

The surface preparations and cleaning procedures had to be limited to those compatible with electronic circuitry, and performance. In other words, drastic chemical methods such as acid etching were omitted in this study. One could hope to remove salt like residues, oily residues and particulates by spraying with 200 proof alcohol or by ultrasonic cleaning

with Freon TF or by vapor degreasing with trichloroethylene or trichloroethane or by a combination of these. One could hope to remove oxides and scale by sand blasting with either glass balls or with Black Beauty "sharp" grit. (FeO 23%, Silica 42.7%, Al_2O_3 21%, Mesh: 50).

Some authors stress the importance of using solvents to dissolve both water-insoluble constituents, the hydrophobics, and water soluble contaminants, the hydrophilics. Table II is taken from J. J. Licari's books⁹ as a classification of solvents types.

TABLE II--CLEANING SOLUTIONS AND SOLVENT TYPES⁹

Chemical Type	Examples
Hydrophobic	
Organic solvents	Naphthas, xylene, toluene
Fluorocarbons	Freon, TF, Freon TMC
Chlorinated hydrocarbons	1,1,1-Trichloroethane, Perchloroethylene, trichloroethylene
Hydrophilic	
Organic solvents	Acetone, methyl ethyl ketone (MEK), methanol, ethanol, isopropanol
Ionic	Alkaline, acid, and detergent water solutions
Nonionic	Detergent-water solutions
Water	Tap, deionized, or distilled
Hydrophobic-hydrophilic	Alcohol naphtha (50:50 mixture), a fluorocarbon-water emulsion containing a surfactant, an azeotrope of fluorocarbon and acetone, a blend of fluorocarbon and ethyl alcohol.

Results

Results of the adhesive strength measurements are shown in the following Tables. Each average is based on six samples:

TABLE III LAP SHEAR STRENGTH OF
SHELL EPON 828/MILLER STEPHENSON V-40

(A) Adherent: 60% tin 40% lead solder, electro-plated on Beryllium Copper.

Adhesive Thickness: 0.019 inches \pm .002

<u>Surface Treatment</u>	<u>Standard Deviation psi</u>	<u>Average Strength psi</u>
As received	± 100	700
200-proof Ethanol spray	± 80	700
Ultrasonic clean, with Freon TF	± 230	1000
Ultrasonic clean Freon, paper towel rub, ultrasonic clean	± 250	1180
Vapor degrease Trichloroethane, 74°C.	± 260	1170
Vapor degrease Trichloroethylene, 84°C.	± 240	1220
Ethanol sprayed, SiC 320 paper by hand, Ethanol sprayed	± 50	1200
Ultrasonic clean "sand blasted" with glass balls, ultrasonic clean Freon TF	± 210	1360
Trichloroethylene vapor degreased, "sand blasted" with glass balls, vapor degreased	± 230	1810

<u>Surface Treatment</u>	<u>Standard Deviation psi</u>	<u>Average Strength psi</u>
Ultrasonic clean Freon, "sand blasted" with Black Beauty grit, ultrasonically cleaned	<u>+120</u>	1950

(B) Adherent: Glass Epoxy Board

Adhesive Thickness: 0.019 inches ± .002

Vapor Degrease with Trichloroethylene	<u>+240</u>	1350
Ultrasonic clean, Freon TF	<u>+100</u>	1900
200- proof Ethanol Spray	<u>+260</u>	2000
Vapor degrease with trichloroethylene, sand blast with glass balls, vapor degrease	<u>+80</u>	1380
Ultrasonic clean Freon TF, sand blast with glass balls, ultrasonic clean	<u>+240</u>	1430

(C) Adherent: Porcelain

Adhesive Thickness: 0.019 inches ± .002

Scrubbed with Bon Ami,
rinsed with distilled water,
dried, vapor degreased trichloroethane

> 1400

Porcelain broke on all samples before adhesion failure.

(D) Adherent: Ferrite

Adhesive Thickness: 0.019 inches ± .002

Ultrasonic clean with Freon TF

> 1130

Ferrite broke on all samples before adhesion failure.

TABLE IV

VARIATION OF LAP SHEAR STRENGTH WITH GLUE LINE THICKNESS

Adhesive: Epon 828/Miller Stephenson V-40

Adherent: Glass Epoxy Board

<u>Thickness of Glue Line Inches</u>	<u>Standard Deviation psi</u>	<u>Lap Shear Strength psi</u>
.008"	+300	2450
.019"	+100	1900
.030"	+290	1710

Adherent: 60-40 Solder on Beryllium Copper

Surface Prep: Ultrasonic clean, Black Beauty Grit Ultrasonic clean

.010"	+50	2100
.019"	+120	1950

Adherent: 60-40 Solder on Beryllium Copper

Surface Prep: Ultrasonic Paper Towel Rub, Ultrasonic

.010"	+100	1460
.019"	+250	1180

TABLE V

LAP SHEAR STRENGTH OF THIOKOL SOLITHANE 113

(A) Adherent: 60-40 Solder, Electro-plated on Beryllium Copper

Adhesive Thickness: 0.010 inches \pm .002

<u>Surface Treatment</u>	<u>Standard Deviation psi</u>	<u>Lap Shear Strength psi</u>
Ultrasonic Clean with Freon TF	+25	90
Vapor Degrease Trichloro- ethane	+15	95
Alcohol Spray	+5	110

<u>Surface Treatment</u>	<u>Standard Deviation psi</u>	<u>Lap Shear Strength psi</u>
Vapor Degrease with Trichloroethane, sand blast with Black Beauty, vapor degrease	+15	160
Vapor degrease with Trichloroethane, prime with thin coat of Epon 828/V-40	+65	355

(B) Adherent: Glass Epoxy Board

Adhesive Thickness: 0.010 inches \pm .002

Vapor degrease with Trichloroethane, grit blast Black Beauty grit, vapor degrease	+30	215
Vapor degrease with Trichloroethane	+40	220

(C) Adherent: Ferrite

Adhesive Thickness: 0.010 inches \pm .002

Ultrasonic clean, hand sanded on 400 grit SiC paper, Ultrasonic clean with Freon TF (Johnson)	+15	60
Repeat above (Clatterbuck)	+20	60
Ultrasonic clean only	+20	100

(D) Adherent: Porcelain

Adhesive Thickness: 0.010 inches \pm .002

Data very poor despite many tries. Perhaps the heavy cutting grease from the diamond saw is never quite removable.

Vapor degreased, Trichloroethane	+30	30
----------------------------------	-----	----

<u>Surface Thickness</u>	<u>Standard Deviation</u>	<u>Lap Shear Strength psi</u>
Vapor degreased Trichloroethane, Bon Ami scrubbed, washed, dried, vapor degreased	<u>+65</u>	75
Ultrasonic clean Freon, Bon Ami scrubbed, washed, dried, ultrasonic clean Freon	<u>+50</u>	80

TABLE VI

LAP SHEAR STRENGTH OF SYLGARD 184. PRIMED WITH SYLGARD PRIMER.

(A) Adherent: Solder

Adhesive Thickness: 0.010 inches + .002

<u>Surface Treatment</u>	<u>Standard Deviation psi</u>	<u>Lap Shear Strength psi</u>
Vapor degreased Trichloroethane, primed with Sylgard primer	<u>+75</u>	320
Vapor degreased Trichloroethane, grit blast Black Beauty, vapor degreased again, primed	<u>+60</u>	495
Same as above, 0,020" glue line	<u>+35</u>	470

(B) Adherent: Glass Epoxy Board

Adhesive Thickness: 0.010" + .002

Vapor degreased Trichloroethane, primed Sylgard primer	<u>+45</u>	315
Same as above and grit blasted Black Beauty grit	<u>+20</u>	565

Conclusions:

The greatest amount of data was obtained with Epon 828 Epoxy as shown in Table III and IV. For solder, the shear strength varies all the way from 700 psi, as received, to 1950 psi when the surface was sand blasted with sharp Black Beauty grit between two sessions of ultrasonic cleaning with Freon TF. Sand blasting preferably with sharp grits, markedly enhances the adherence to solder. But even without sand blasting, ultrasonic cleaning with Freon or vapor degreasing with Trichloroethane or Trichloroethylene seems to produce better bonding than alcohol spray.

On glass epoxy board, strangely, the reverse seems true. The best bonding is obtained with just plain ethanol spray. Sand blasting makes for less strong bonding. Perhaps this can be explained on the basis that the epoxy of the glass epoxy board sticks best to the Epon 828 epoxy when its surface is not disturbed enough to expose the glass fibre or attacked by active solvents such as Trichloroethylene.

The ferrite and porcelain materials always broke before the Epon adhesive joints failed. Adhesive data to porcelain and ferrite thus shows that even to these brittle materials epoxy will adhere very well, so that lap shear strength is well above 1000 psi.

Table IV is included to reinforce the well known fact that, in general, the thinner the glue line the greater the measured lap shear strength. This is partially due to the negligible peel torque for thin joints and also partially

due to the fact that many adhesives simply "like" a thin glue line.

The measurements with Solithane 113, which is a polyurethane, are somewhat disturbing. As Table V shows, the adhesive strength was generally weak, around 100 to 200 psi except when Epon 828 was used as a thin primer coat, when 355 psi was achieved. Again, sand blasting enhanced the adhesion to solder, but did not affect adhesion to glass epoxy board.

Because the adhesive strength of Solithane was weak, variations of surface preparation could now be done on ferrite. In other words, the adhesive joints failed before the ferrite broke, thus revealing influence of surface preparation. Here it was interesting to observe that sand blasting or sanding on SiC paper had just the opposite effect of what was intended. It actually polished the ferrite surface due to producing a layer of fine ferrite dust which acted as polishing powder. The adhesive strength thus was decreased.

The porcelain data with Solithane is the least satisfactory despite many tries. Unfortunately, when reasonably priced porcelain plates of suitable thickness, were finally located, so that they were not so thin as to break and not so thick as not to fit into the tensile tester grips, they were too wide. They had to be sawed in half on a diamond saw, and the heavy cutting grease was afterwards not completely removable by any of the cleaning solvents. Therefore, it is thought, the data on porcelain sets of 6 scatter all over the place as shown in Table VII~~2~~.

TABLE VIIa

CONTRAST IN DATA SCATTER FOR A GIVEN SET OF 6 ADHESIVE SAMPLES,
FOR A GLASS-EPOXY SET AND A PORCELAIN SET.

	<u>Shear Strength psi</u>
Sylgard 184 on glass-epoxy after grit blasting and vapor degreasing	550 586 560 600 548 <u>548</u>
Average	565 ± 20
Solithane 113 on porcelain after scrubbing with scouring powder, then ultrasonic clean Freon TF	145 16 123 96 <u>21</u>
Average	80 ± 60

The Thiokol Corporation recommends Woolsey Metallast 919/920 as primer for Solithane 113. This was not used because, according to the Materials section, GSFC, it outgases badly and also contains phosphoric acid. It is encouraging that with use of epoxy primer the adhesive strength of Solithane 113 to solder did reach into the 300's of psi.

By contrast, the Sylgard data in Table VI was surprising. Sylgard primer was of course always used. Grit blasting with Black Beauty grit increased the bonding strength to solder and to glass epoxy board, to around 500 psi. This is greater than

achieved with Solithane 113 so far, even though silicone rubbers have the reputation of poor adhesion. Correspondence with the Thiokol Corporation is now in progress to see what information they can supply on adhesion of their Solithane 113.

Suggestions for Potting of Electric Circuits:

(1.) From the point of view of improved adhesion, it would seem that sandblasting with a fine quartz grit would be advantageous. From the electrical point of view, however, the question arises whether any remnants of the grit or of blasted, loose metal particles would cause electrical noise. The author would, therefore, suggest thorough testing of electric noise level with and without grit-blasting before recommending grit blasting.

(2.) A sequence of solvent cleaning certainly suggests itself.

A sequence such as:

- (1) Rinse in distilled water
- (2) Dry thoroughly
- (3) 200-proof Ethanol spray
- (4) Dry
- (5) Ultrasonic clean in Freon TF or just dip--clean in Freon TF or vapor degrease in trichloroethane (74°C = 165°F)

would seem to be a more complete cleaning than only an alcohol spray. (The Freon TF used would, of course, have to be pure and particle-free).

(3.) Since likelihood of separation of the electric components from the potting compound is less for greater adhesive strength, but more for greater internal stresses, these internal stresses have to be minimized. An attempt to study the internal stresses

due to the initial polymerization and due to temperature cycling is now in progress.

(4.) In view of the seemingly poor adhesion of Solithane 113 as measured to-date, there seems to be no advantage to potting with Solithane 113 rather than with Silicone rubber.

CHAPTER II PERMEATION STUDY

Introduction

The high voltage portions of electric power supplies must be encapsulated in "potting" materials to prevent electric breakdown. Air bubbles or air in hollow core resistors may inadvertently be trapped in the solid potted module, and the question then arises as to how long it will take for this air to diffuse out into the vacuum of space until the pressure in the bubble decreases to 10 torr to 0.1 torr. When this region of pressures, the corona region, is reached, then electric breakdown is probable across the bubble if it is close to a metallic electrode, even for very modest potential differences such as 400 volts. To calculate how much time will elapse before the corona region of pressures is reached requires a knowledge of the permeation constants of air through the potting polymers. The permeation constant depends on temperature, of course, but since the excursion of temperatures of equipment in a scientific satellite is very little above 25°C (only about 10°C to 20°C) it was felt that measurement at that ambient temperature would give representative results.

Method:

In Materials--Science Laboratories, methods using a polymer permeation analyzer or a Barrer-type permeation cell are used for very thin membranes.¹⁰ Since the project first began in a high voltage electronics laboratory where such equipment was not available or was not easily purchased, a different approach was chosen.

Method A1: The polymer in question is molded in the shape of a cylinder with a cavity inside. Only one wall is thin, in case of the cube--one face, in case of the cylinder--the cylindrical wall. (See Figure 3) The thicknesses of the other walls and all possible interfaces to other materials are at least one order of magnitude larger. Electrodes of 1/8" diameter stainless steel are embedded in the polymer such that they extend into the cavity, and the gap between them is spaced about one millimeter apart. A thick walled glass tube is also sealed in for attachment to a vacuum system. For each cylinder, a calibration curve is obtained of breakdown voltages of the 1 mm gap versus gas pressure in the cavity, using a thermocouple type gauge from 0.01 to 1 mm Hg and a mercury manometer from 2 mm to 760 mm. Figure 4 shows a typical calibration curve. The unit is then sealed off at 0.01 mm pressure and the rise in internal pressure with time is monitored by determining the

breakdown voltage of the gap at suitable time intervals, at first every two hours and later once a day, or once a week, depending on behavior.

Method A2: In order to verify the above measurements, two Epon 828 and one Stycast 3050 cylinder had Veeco Type 4M thermocouple tubes sealed in the top instead of the spark gap electrodes. Thus internal gas pressures up to 20 torr could be read directly as a function of time. For pressures above 20 torr, these tubes are, however, not sensitive.

Method B: The hollow cylinders were sealed off with atmospheric pressure air trapped inside the cavity. These were then placed in a vacuum system at 10^{-5} torr pressure for long periods of time. The goal was to find how much time would be required for the inside pressure to drop to a low value due to outward permeation of gas, again inferred from breakdown voltages of the spark gap.

Theory:

The diffusion constant D in cm^2 /second is defined by Fick's

Law 11, 12 $J = -D \nabla C$ (1)

which in the one dimensional case becomes

$$J = -D \frac{\partial C}{\partial x}$$

where

J = number of diffusing atoms flowing per cm^2 per second perpendicularly through an area in the material.

C = number of diffusing atoms per unit volume in the material

$\partial C / \partial x$ = concentration gradient of gas atoms at any point within the material.

t = time in seconds.

The number of diffusing atoms is conserved and thus the continuity equation follows from (2).

$$\frac{\partial C}{\partial t} = D \nabla^2 C$$
 (3)

which in the one dimensional case becomes

$$\frac{\partial C}{\partial t} = \frac{D \partial^2 C}{\partial x^2}$$
 (4)

Now, if one (a) considers diffusion through one wall of the void where one only measures concentrations just beyond each boundary and if one (b) converts from number of atoms to volume of gas and resulting pressure changes and if one (c) considers the pressure near one boundary to be practically zero, $p_2 = 0$ and near the other boundary to be atmospheric, $p_1 = 1$ atmosphere, such that the pressure gradient $\frac{p_1 - p_2}{L}$ is almost constant, then

the exact solution¹³ of equation (4) is:

$$\frac{\Delta p}{\Delta t} = \frac{A \cdot D \cdot S (p_1 - p_2)}{V_{app} L} \left\{ 1 + \sum_{m=1}^{\infty} 2 \cos^2 m \exp\left(\frac{-m^2 \pi^2 D t}{L^2}\right) \right\} \quad (5)$$

where

Δp = pressure change inside void due to diffusion

p_1, p_2 = outside, inside gas pressures

L = thickness of wall

A = cross section area of wall.

S = solubility of gas in wall material

V_{app} = volume of cavity

$D.S = \underline{P}$ = permeation constant

After a long enough time this becomes

$$\underline{P} = \frac{V_{app} \cdot L \cdot \Delta p}{A (p_1 - p_2) \Delta t}$$

Convert from atmosphere to cmHg and correct for STP, then at 27°C

$$\therefore \underline{P} = \frac{V_{app} \cdot L \cdot \Delta p}{A (p_1 - p_2) \Delta t} \cdot \frac{273^\circ}{300^\circ} \cdot \frac{76 \text{ cmHg}}{1 \text{ atm}} \quad (6)$$

$$\text{in } \frac{\text{cm}^3(\text{STP}) \cdot \text{cm}}{\text{cm}^2 \cdot \text{sec} \cdot \text{cmHg}}$$

Equation (6) holds equally well for rectangular geometry as for cylindrical geometry in the approximation that the thickness of the cylindrical wall is small compared to the mean radius of the cylinder¹³.

If p_2 changes slowly with time, then approximately

$$p = 76 [1 - \exp(-\frac{Pt}{Lk})] \text{ for } p_2 = 0 \text{ at } t = 0; p_1 = 76 \text{ cmHg} \quad (7)$$

$$\text{or } p = 76 \exp(-\frac{Pt}{Lk}) \text{ for } p_2 = 76 \text{ cmHg, } t = 0; p_1 = 0 = \text{const.} \quad (8)$$

where $k = V_{app} \times 273 / (A \times 300 \times 76 \text{ cmHg})$

The equations (7) and (8) are oversimplified and assume that a quasi-steady-state situation exists. In actuality, this is not true since the concentration gradient is changing as p in the cavity changes. But if this is slow enough, then equations (7) and (8) should yield fair approximations of the time required for the pressure in the cavity to reach certain values.

Most investigators^{10,14,15,16} measure \underline{P} from equation (6) using steady state flow, measure D from the transient time t_c required to establish the steady state and then solve for solubility S . Our method is not sensitive enough to obtain D from the short time approximation for t_c , due to outgasing, and we can only measure \underline{P} in the long-time steady-state situation.

Results:

As stated earlier in Method A1 each permeation cylinder had its own calibration graph of breakdown voltage versus pressure. Figure 4 is one example, and it is thought of little interest to the reader to reproduce all the calibration curves. From these, then, the curves of pressure versus time of the cylinders were obtained.

(A) One can begin with Stycast 3050, measured as above in Figure 6, and measured by thermocouple pressure gage Veeco 4M in Figure 7. Both cylinders show an almost immediate rise in pressure after sealing off due to outgasing. Thereafter, the rise in pressure is very, very slow and can be blamed on permeation through the thin wall. The slopes are variously $\frac{0.4 \text{ torr}}{\text{month}}$, $\frac{3 \text{ torr}}{9 \text{ months}}$ and $\frac{1.3 \text{ torr}}{3 \text{ months}}$ which gives an average

of $\frac{.38 \text{ torr}}{\text{month}}$. This can be substituted in equation (6)

$$\begin{aligned} P &= \frac{V_{app} \times 273 \times \Delta p \times L}{A \times 300 \times \Delta t \times (p_1 - p_2) \times 76} \\ &= \frac{32 \text{ cm}^3}{21 \text{ cm}^2} \times \frac{273}{300} \times \frac{.038 \text{ cmHg} \times 0.24 \text{ cm}}{(30.5 \times 24 \times 3600) \text{ sec} (76 \text{ cmHg})^2} \\ &= 0.85(+0.15) \times 10^{-12} \frac{\text{cm}^3}{\text{sec}} \cdot \frac{\text{cm}}{\text{cm}^2} \cdot \frac{\text{cm}}{\text{cmHg}} \end{aligned}$$

(B) Much effort went into the study of the Epon 828 epoxy, and yet the results are not too precise. The basic reasons are the following:

- (1) The Epon 828 outgasing took place much more gradually than in the case of Stycast 3050, and one could therefore not definitely tell where outgasing ended.
- (2) Firing of the spark gap seemed to liberate some gas, such that the more often the spark gap was fired the more steeply the pressure curve rose.
- (3) Using the thermocouple gauge promised to be a way to avoid the above difficulty. But the leveling off of the outgasing occurred within a few torr of the upper limit of sensitivity of the thermocouple gauge tube Veeco 4M. Accuracy was poor near the upper range of the calibration, between 15 to 20 torr, and so the data shown in Figure 10 is not too significant.

Slopes taken from Figures 8 and 9 where the spark gap method of measuring pressure was used is tabulated in Table VIII.

TABLE VII b: Epon 828 Permeation Data

	V cm ³	A cm ²	L cm	$\frac{\Delta p}{\Delta t}$, no firing	$\frac{\Delta p}{\Delta t}$, while firing	Average	Calculated P
Cubic Unit	9.2	4	0.152	16 torr/10 months	14 torr/5 months	2.2 $\frac{\text{torr}}{\text{month}}$	$4.6 \times 10^{-12} \pm 30\%$
Cylinder #3	28	17	0.24	10 torr/9 months	10 torr/4 months	1.8 $\frac{\text{torr}}{\text{month}}$	$4.1 \times 10^{-12} \pm 50\%$

Thus one can conclude for Epon 828 epoxy that:

$$\underline{P} = 4.0(+1.5) \times 10^{-12} \frac{\text{cm}^3 \cdot \text{cm}}{\text{sec} \cdot \text{cm}^2 \cdot \text{cmHg}}$$

This is much lower than the preliminary value reported earlier¹⁷. During the brief two or three weeks of observation at the earlier time the Epon 828 was still outgasing internally into the evacuated cavity; also there was a tendency to fire the spark gap too often.

Both types of epoxy, Stycast 3050 and Epon 828 were also investigated by Method B. Atmospheric pressure was trapped inside and the units were placed in a vacuum system at about 3×10^{-6} torr with 3 kv continuously on the spark gap. No breakdown occurred during a period of 73 days. When a variable voltage was applied, the Epon 828 broke down at 5.26 kv, which was within the range of calibration voltages at atmospheric pressure and the Stycast broke down at 7.5 kv, 0.75 kv higher (!) than calibration voltage at atmospheric pressure! In other words, negligible amounts of gas had diffused out into the vacuum.

(C). Figure 11 shows the permeation history of Solithane 113, cylinder #2. It is obviously much faster than for the epoxies, the pressure changing by one-half atmosphere inside a four month interval. Obviously, the diffusion slows down as the pressure gradient between inside and outside decreases. One could use equation (7) to calculate \underline{P} , or use the initial slope of the curve, while the concentration gradient was still essentially constant, in the normal equation (6). The latter is chosen here, using $V = 32 \text{ cm}^3$, $A = 21 \text{ cm}^2$, $L = 0.24 \text{ cm}$, $\Delta p / \Delta t = 110 \text{ torr} / 14 \text{ days}$ from Figure 11 and $110 \text{ torr} / 10 \text{ days}$ from Figure 12.

Also 70.5cm is used for the average $(p_1 - p_2)$. This results in 5.5×10^{-10} and 7.5×10^{-10} , with an average $\underline{P} = 6.5(\pm 1.0) \times 10^{-10} \frac{\text{cm}^3 \cdot \text{cm}}{\text{cm}^2 \cdot \text{sec} \cdot \text{cmHg}}$ for Solithane 113. The Solithane is flexible. Thus a nylon liner with holes had to be used inside the cavity wall to keep it from collapsing. The larger holes were in the Solithane unit #4, which might explain the larger \underline{P} calculated for this unit, less likely to obstruct the free access of gas molecules to the inner wall.

(D). The actual silicone rubber polymer used for potting flight hardware is Dow Corning 93-500 and is extremely expensive. In its place Dow Corning Sylgard 185 was used for the diffusion study as a closely related compound.

Figure 13 is a composite graph, showing data points of several permeation runs on the same unit and on two different units. The permeation was quite fast, so that pressure changed from vacuum to atmospheric inside the cavity within 4 days. A nylon liner with holes had to be used inside these flexible cylinders, just as with the Solithane. The permeation constant can again be calculated from the slope at the beginning of the pressure versus time curve. This yields $\frac{190 \text{ torr}}{6 \text{ hours}}$ and $\underline{P} =$

$50 \times 10^{-9} \frac{\text{cm}^3 \cdot \text{cm}}{\text{cm}^2 \cdot \text{sec} \cdot \text{cmHg}}$ Or, one can plot the data of (76-p)

versus t as demanded by equation (7), which yields a straight line, on semilogarithmic paper.

$$p = 76 \left[1 - \exp \left(-\frac{Pt}{Lk} \right) \right] \quad (7)$$

$$\ln (76 - p) - \ln 76 = - \frac{pt}{Lk}$$

This is a straight line whose slope is:

$$2.303 \frac{\Delta \log (76-p)}{\Delta t} = - \frac{p}{Lk} \quad (9)$$

$$\text{where } k = \frac{V_{app}}{\text{Area}} \times \frac{273}{300} \times 76 \text{ cmHg}$$

The Sylgard 185 data is plotted according to equation (9) in Figure 14.

From this graph the slope is

$$\frac{\log 7.6 - \log 76}{62 \text{ hours}}, \text{ which gives}$$

$$\text{for } p = \frac{2.3 \times 1 \times .24 \times 32 \times 273}{62 \times 3600 \times 21 \times 300 \times 76}$$

$$p = 46. \times 10^{-9} \frac{\text{cm}^3 \cdot \text{cm}}{\text{cm}^2 \text{ sec cmHg}}$$

The Sylgard permeation could also be run in the reverse direction sealing one atmosphere into the cavity and causing pressure to drop from 760 torr to 60 torr in 3 days and 5 hours, or 77 hours. For this the slope is $\frac{\log 6 - \log 76}{77 \text{ hours}}$,

which from equation (8) calculates into

$$p = 41. \times 10^{-9} \frac{\text{cm}^3 \text{ cm}}{\text{cm}^2 \text{ sec cmHg}},$$

giving an average for Sylgard 185

$$P = 43(+3) \cdot 10^{-9} \frac{\text{cm}^3}{\text{cm}^2} \frac{\text{cm}}{\text{sec cmHg}}$$

The Sylgard 184 permeation was somewhat less, namely
 24×10^{-9} for diffusion from outside air into the cavity and
 34×10^{-9} from data for the reverse process, giving an average
of about $30(+5) \times 10^{-9}$ for Sylgard 184. No repeat experiments
were performed on Sylgard 184.

Conclusion:

Table VIII summarizes the measured results for the four types of potting compounds. One can conclude that using a relatively simple method, the permeation constants were obtained to one--significant figure accuracy, which certainly permits one make to "ball park" calculations such as suggested in the introduction. Also, a reference was found during library search for adhesion reports, that cites permeation constants for various types of polymers at ambient room temperature¹⁸ . It is reassuring that for the same types of polymers in that reference the same order of magnitude is reported as the measured results in this present study.

TABLE VIII PERMEATION RESULTS

<u>Name of Polymer</u>	<u>Type</u>	<u>Measured P in this Study</u> $\frac{\text{cm}^3 \cdot \text{cm}}{\text{cm}^2 \cdot \text{sec. cmHg}}$
Stycast 3050	Epoxy	$0.85(+.15) \times 10^{-12}$
Epon 828	Epoxy	$4.0(+1.5) \times 10^{-12}$
Solithane 113	Polyurethane	$6.5(+1.0) \times 10^{-10}$
Sylgard 185	Silicone Rubber	$43(+3) \times 10^{-9}$
Sylgard 184	Silicone Rubber	$30(+5) \times 10^{-9}$

Some P Values for similar Types from Major and Kammermeyer¹⁸

Epon 1001, for O_2 , 4.9×10^{-12}
Epoxy

Estane, for O_2 , 1.7×10^{-10}

Polyurethane, for O_2 , 2.1×10^{-10}
PC6

SiLastic, Si-rubber, for air, 33×10^{-9}

RTV 90, Si-rubber, for O_2 , 53×10^{-9}

RTV 502, Si-rubber for O_2 , 36×10^{-9}

One can now make an order of magnitude calculation for the length of time for pressure in a bubble to drop from 760 torr to 10 torr in the vacuum of space. Assume a cubic "bubble", 1cm under the surface of the polymer, 3.3mm on a side, and use equation (8).

TABLE IX

Time for Pressure in Bubble to drop from 760 to 10 Torr.

<u>Polymer</u>	<u>t</u>
Stycast 3050	2.3×10^5 days = 620.years
Epon 828	130.years
Solithane 113	300.days
Sylgard 185	4.26×10^5 sec = 5 days

Finally, based on this, what should one do? If bubbles are trapped in either type of epoxy, at atmospheric pressure, they will not drop to corona pressures for years and years, and so they will not break down catastrophically. If they are nowhere near a metal electrode they will probably not contribute occasional tiny noise counts. They need not be a source of worry, and need not be repaired.

In the case of Solithane and certainly in the case of Silicone rubber the permeation of air is much faster, corona pressures could be reached in a matter of days or months and any trapped bubbles should be cut into, removed, and filled in solidly.

CHAPTER III

Behavior of the Series 600--Type "L" Cable-High Voltage Connectors, at Various Pressures.

MANUFACTURED by REYNOLDS INDUSTRIES, INC., 5005 McConnell Avenue, Los Angeles, CALIFORNIA 90066.

The Reynolds Series 600 high voltage connectors are internally completely clad by diallyl phthalate. In addition, both the male and female connectors have small internal O-rings in an attempt to make them leak-proof. When the male and female connectors are tightly mated, the diallyl phthalate shoulder of the female connector is jammed up against the O-ring of the male such that there is essentially no easy gaseous path for charged particles from the vicinity of the high voltage center pin to travel to the grounded outer metal housing of the female connector. Thus catastrophic discharge is avoided, even in the corona region of pressures, which for these connectors lies between about 1.5 torr to 10 torr. Figures 15 and 16, the latter copied with permission from the Reynolds company, show the geometry and essential parts of the P/N 167-3771 male panel connector and P/N 167-3770A female cable connector with "L" type cable.

The experimental set-up for testing was largely designed and assembled by Dr. John F. Sutton of GSFC, Code 325.1 as shown in Figure 17. The connector under test is the one in the vacuum chamber. Originally, Reynolds connectors and cables were used

between points AB, BD and BE outside of the vacuum chamber. Under these conditions the circuit was electrically noisy, so as to obscure the behavior of the test connector in the vacuum chamber. Potting the 10 megohm resistor at A and the 0.001 uf condenser at D gave no improvement. However, the following steps did improve the performance:

- (a) Replacing the never--outgased Reynolds connector and cable between points AB and BD by solid pieces of buss wire.
- (b) Replacing the never-outgased Reynolds connectors and cables between points BE and F with an RG-59 cable--high vacuum feed through. This was continuous from points B to P where it was spliced onto the Reynolds type "L" cable of the connector under test. This solder joint was solid potted with Epon 828 epoxy. But the stripped--back shielding braid and teflon outer insulation was left untouched by the epoxy, and thus, that end of the Reynolds "L" type cable was open and not sealed. Three inches of "L" type cable were used for the connector--testing.
- (c) The vacuum chamber with contents was outgased at 10^{-3} torr for at least 24 hours.

The above steps resulted in zero count-rate at 10^{-3} torr and gave a starting point for the investigation.

TEST I: Variation of Count Rate with Pressure.

Sensitivity: All counts from 3×10^{-14} coulombs on up, window counter 3×10^{-14} to 2×10^{-13} coulombs. The latter counts generally comprised about 80% of all counts. The applied voltage was 3000 volts.

A great deal of data was taken over a span of about a month with two different female connectors, the first assembled by Reynolds, the second at GSFC. It must be remembered that the connector was filled with and surrounded by gas at the pressures shown in Figures 18a to 24c. Sometimes different inside pressure p_i was used than outside pressure p_o . The corona region was determined easily by slightly unscrewing the connectors. If the counts increased by several thousand in an "instant", then that meant corona region pressures, and the thousands of counts per instant represented corona discharge. For these connectors the corona region was between 1.5 to 10 torr. A summary of Test I results is as follows:

- (1) After outgasing at 10^{-3} torr for 2 days the Reynolds connectors and 3" length of "L" type cable (one end open) are noise free below and above the corona region.
- (2) In the corona region, the connectors and 3" of "L" type cable never broke down catastrophically, even after innumerable openings and closings.
- (3) In the corona region, when filled with and surrounded by gas at corona region pressures, the connectors did give some extra noise counts. The commercially assembled connector gave about 20 counts/5 minute interval and the one assembled at GSFC

about 10 counts/5 minute interval. The latter one had the cable end sealed with Stycast 3050, where it interfaced with the connector proper.

(4) The noise counts appeared in two types of modes: Either a steady dribble of single counts or a period of complete quiet for several minutes followed by a multi-count burst. The latter behavior occurred most often after long outgasings. Observing the pulses at the output of the operational amplifier at O with a Tektronix "memory" oscilloscope it became evident that the multiple bursts were very energetic single pulses, followed by high voltage shock oscillations in the circuit. Substituting a filter network,* for the single 0.001 uf capacitor at D, during subsequent experiments, eliminated the multiple burst. (*Essentially 7 channels of a 250M resistor in series with a 100M resistor in series with a 1100 pf, 4Kv capacitor. Between the two resistors another 1100 pf, 4Kv capacitor goes to ground.)

TEST II:

New male and female connectors were installed to do a long-term test in the corona region. That is, the connector was filled with and surrounded by gas at pressures of 2 torr, the valve to the vacuum pump was turned off and then 2500 volts was applied and the system left this way day and night. Every second or third day the total count was noted and average count rate computed from this in counts/5 minute interval. Since the pressure by that time had usually crept up to 8 or 10 torr, the voltage was momentarily turned off, the system pumped down to 2 torr again, the connector reopened and reclosed, the voltage turned back on and the count rate observed for about 20 minutes

subsequently. Then the system was left this way again for 2 or 3 nights and days, and so on. An abbreviated data log follows in Table X. (Obviously, the female connector had to have 3" of "L" type cables on it again, as before, splice potted, braid sealed at one end with Solithane 113, open at the other end).

TABLE X: Abbreviated Data Log

Long-time test in corona region of Series 600 Reynolds Connectors,
3" of type "L" cable attached. Continuously in Corona region:
1.5-10 torr.

Discriminator setting: Lowest level = 8×10^{-15} coulombs on up.

Window counter: 8×10^{-15} coul to 8×10^{-14} coul

Voltage applied: 2500 volts

Filter network installed in place of condenser at D.

Filter network alone,

test chamber out of circuit 5cts/5 minutes

Outgas until

October 29, 1974 - Begin

p = 2 torr, V = 2500 volts

Count is 13.5/5 minutes, F.5/5 minutes

October 31, 1974

p = 4 torr, V = 2500 volts

Count is 8/5 minutes, 10/5 minutes

November 2, 1974

p has gone up to 7.5 torr by now

Count is 10/5 minutes, 8/5 minutes, 12/5 minutes

Pump p down to 4 torr, open connector, reclose, take count-rate again = 6 counts/5 minutes.

November 5, 1974

p = 9 torr by now

Av. count rate in 3 days = 10.5 counts/minute

Reopen, pump down to 3 torr, close

count rate = 7 counts/minute

November 7, 1975

p = 7 torr

Av. count rate during interval of 2 days = 9 counts/5 minutes

Count rate while watching is = 14/5 minutes, 8/5 minutes

Repump down to 3 torr

count rate = 7, 13, 8/5 minutes

Test interrupted November 8, 9 for floor cleaning!!!

November 12, 1974. Begin again

p = 3 torr

count rate = 6 counts/5 minutes

November 15, 1974

p up to 7 torr

count rate = 8 counts/5 minutes

Repump down to 3 torr

November 19, 1974

p up to 8 torr

Av. count rate during 4 days = 7.5/5 minutes

pump down to 3 torr

count rate = 7/5 minutes

November 21, 1974

p up to 7 torr

Av. interval rate = 8 counts/5 minutes

count rate while watching = 4.5/5 minutes

pump down to 3 torr

count rate = 5/5 minutes

November 26, 1974

Came in at 11:20 a.m.

Count rate way up = 67, 32, 23, 40/5 minutes!!!!

At 12:05 p.m. this all stopped. Some one went to lunch

Probably was interference of some sort.

Now count rate quite low = 2, 3/5 minutes

Pump down to 3 torr.

November 29, 1974

p = 9 torr now.

Av. count rate during last 3 days = 13/5 minutes

Watched count rate = 6/5 minutes

Pump down to 2 torr.

December 3, 1974

p = 9 torr

Av. count rate during last 4 days = 10/5 minutes

Watched count rate = 6/5 minutes

Pump down to 2 torr.

December 7, 1974

p = 7 torr

While standing there, watching from 5:27 p.m. to 6:17 p.m.,
rate went from 8 cts/5 minutes up to 57 ct. /5 minutes and back
down to 6 cts/5 minutes. Must be some sort of interference
(On Saturday evening??)

Steady now at 4, 6, 5 counts/5 minutes

Pump down to 3 torr

December 9, 1974

Av. count rate during last 2 days = 13 cts/5 minutes

December 10, 1974

p = 8 torr

count rate = 11, 8, 10, 8, 8 cts/5 minutes

Back down to p = 3 torr.

December 13, 1974

p = 8 torr

Av. count rate during test 3 days = 10 cts./5 minutes

Pump down to 3 torr

December 20, 1974

p gone beyond 10 torr

count rate during 7 days = 12.5 cts/5 minutes

Pump back down to 3 torr

count rate now only 5, 4, 5, 5/5minutes

Is high voltage getting to connector??

Slowly opened connector while high voltage on. Thousands of counts in a second or so. Yes, high voltage is certainly on!!!

Closed connector.

Count rate now 8, 13 cts/5 minutes

Stopped test. Started working on vacuum system to arrange for gas leakage test.

Conclusion: During 47 days of continuous operation of connector at 2500 volts, while filled with, and surrounded with gas in corona region, 1.5 - 10 torr, there was no significant steady increase in noise count, no tracking damage and certainly no catastrophic breakdown.

TEST III:

A further question was raised as to the behavior of "L" type Reynolds cable, cable alone. Earlier, already, several cables 1 ft. long were noise-tested at atmospheric pressure, at 3000 volts, C=0.001 uf at D rather than the filter, sensitivity 3×10^{-14} coulombs on up, results shown in Table XI.

TABLE XI

One foot of cable noise test at atmospheric pressure.

3000 volts, no filter, sensitivity 3×10^{-14} coulombs.

<u>Cable Type</u>	<u>Counts/10 minutes</u>	<u>Counts/hour</u>
Teledyne Thermatics, formerly Brand-Rex Cat # 60HF, Size 26 gage, M 81381/1 <u>rated at 1 kv,</u> <u>Shielded</u>	266/5 minutes 211/5 minutes	2880/hour!!
Reynolds Type "L" Rated at 15 kv shielded	10/10 minutes 16/10 minutes	78/hour
Raychem Corp. 4H/0511-20-2 rated at 1 kv, not shielded	10/10 minutes 10/10 minutes	60/hour
Harvey Industries 26 . 020 HF 7/34 ShF P/N-30-01938 rated at 10 kv, shielded	6/10 minutes, 8/10 minutes	42/hour
Bare Buss Wire No shielding	2/10 minutes 4/10 minutes	12/hour
RG 59 Cable Shielded	2/10 minutes 2/10 minutes	12/hour

Three feet of Reynolds type "L" cable were spliced onto the RG 59 cable in the vacuum system, and the splice and other end potted with Epon 828, but shielding braid not sealed and thus open for rapid outgasing. The system was then evacuated, 3000 volts applied to the cable. During the act of the first pump-down, there was a very large number of noise counts. This had been observed before when other connectors and cables were first pumped down. When the vacuum valve was turned off during this rapid counting the count rate became zero, only to begin again when pumping was resumed. It was concluded, therefore, that the noise was probably due to charged particles of water vapor being pumped off rather than due to a sustained corona discharge within the cable, at a particular pressure. In fact, the attempt to keep the system at that pressure by rapidly closing the valve resulted in stopping the noise count. Moreover, letting the system up to atmospheric pressure and pumping down again always gave a much reduced noise count. For the 3 ft. cable we had: 23,000 counts during first pump down, about 1200 counts during the next pump-down.

Subsequent tests on this cable had to be done with the filter circuit installed at D instead of the 0.001 uf capacitor. This was because with the 3 ft. length of cable in the circuit, it was easily thrown into oscillations. The sensitivity was set at 8×10^{-15} coulombs and up. Under these conditions the cable was quiet at 0.1 torr and had variously between 0 and 6 counts/5 minutes interval in the corona region from 1 to 10 torr. More work should be done with sealed cable (sealed according to directions by the Reynolds Manufacturing Company, Figure 16).

TEST IV

It is of interest to learn at what rate gas leaks out into the vacuum of space from the series 600 connectors, when mated. This can be done by two methods.

A: Open the connector in the vacuum jar while it is up to atmospheric pressure. Mate it. Pump down to as good a vacuum as possible. Wait for the desired length of time t . Valve off the jar and immediately open the connector while observing the sudden increase of pressure on a pirani gage. With a connector assembled at GSFC that had been opened and closed many, many times, this gave a change of pressure Δp of 8 torr when t was 1/2 hour and a change Δp of 3 torr for $t = 5$ hours and no discernible Δp for $t = 16$ days. The volume of the vacuum jar was about 1000cc. Thus the 8 torr change meant that the internal volume of the mated connector, V_{con} , is about 0.01cc, which agrees with information from the Reynolds Company. Although the attempt to discern Δp upon opening becomes more and more inaccurate the more gas has leaked out, we can certainly say that most of it leaked out within the 16 days.

B: A much faster method is to attach a Helium leak detector to the vacuum jar. This must be calibrated with a standard leak. Fill the jar up to atmospheric pressure with Helium, while the connector is open. Pump down for 2 hours, then read the apparent leak rate due to remaining residual Helium in the jar. Again fill the jar up to atmospheric pressure with Helium, mate the connector, pump down for 2

hours again, read the leak rate again. The difference of the two leak rates is due to the mated connector leaking He into the jar. For the same connector as in Method A we thus obtained a leak rate of 1.3×10^{-7} atmospheric cc/second. Using the equation that $p = 76 \exp \left(- \frac{\text{Leak rate} \cdot t}{V_{\text{con}}} \right)$ one can calculate that it would take 8 days to leak down to 10 torr and 12.5 days to leak down to 1 torr. Hence, there would be 4.5 days duration within the corona region.

Obviously many more connectors need to be tested this way, both assembled by GSFC and by the Reynolds Company.

Conclusion:

As stated in the Abstract, the Reynolds series 600 connector with "L" type cable suppresses electric discharge in the corona region of pressures. It also has low noise count in the corona region, but is not absolutely noise free. The noise count is between 100 to 200 counts per hour at a sensitivity from 3×10^{-14} coulombs on up at 3000 volts without filter, or the same at a sensitivity from 8×10^{-15} coulombs on up at 2500 volts with filter. About 80% of these counts lie within the lowest decade of sensitivity. Below the corona region the connectors appear noise free.

More tests are planned on gas leakage rate from many connectors. More electric noise studies might be done on "L" type cable alone, of various lengths, at various pressures. Moreover, equipment lends itself to capacitor testing. By inserting various capacitors in place of the 0.001 uf one at D, one could select out quiet from noisy ones. (For this, the test portion in the vacuum jar would of course be disconnected.)

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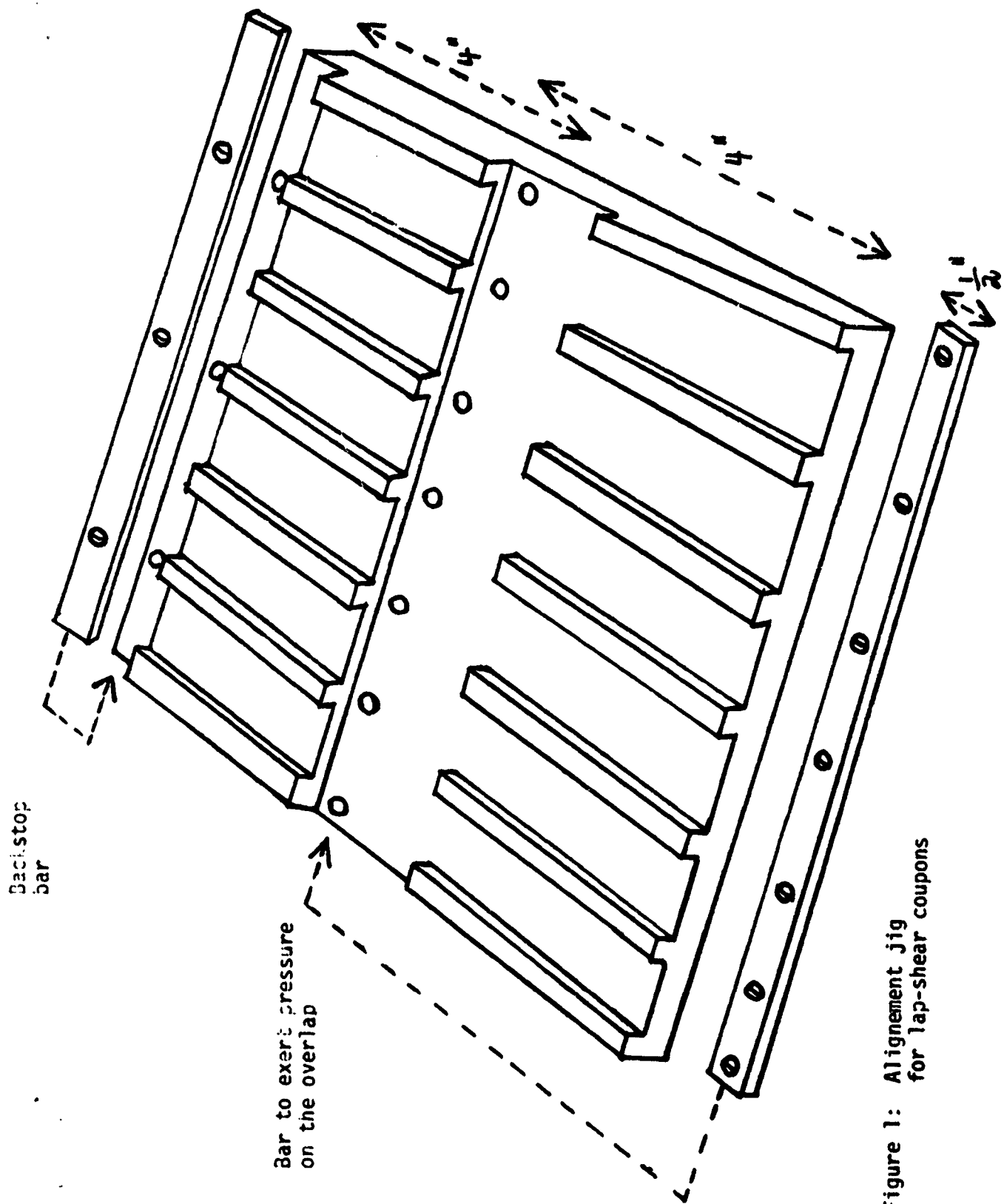


Figure 1: Alignment jig for lap-shear coupons

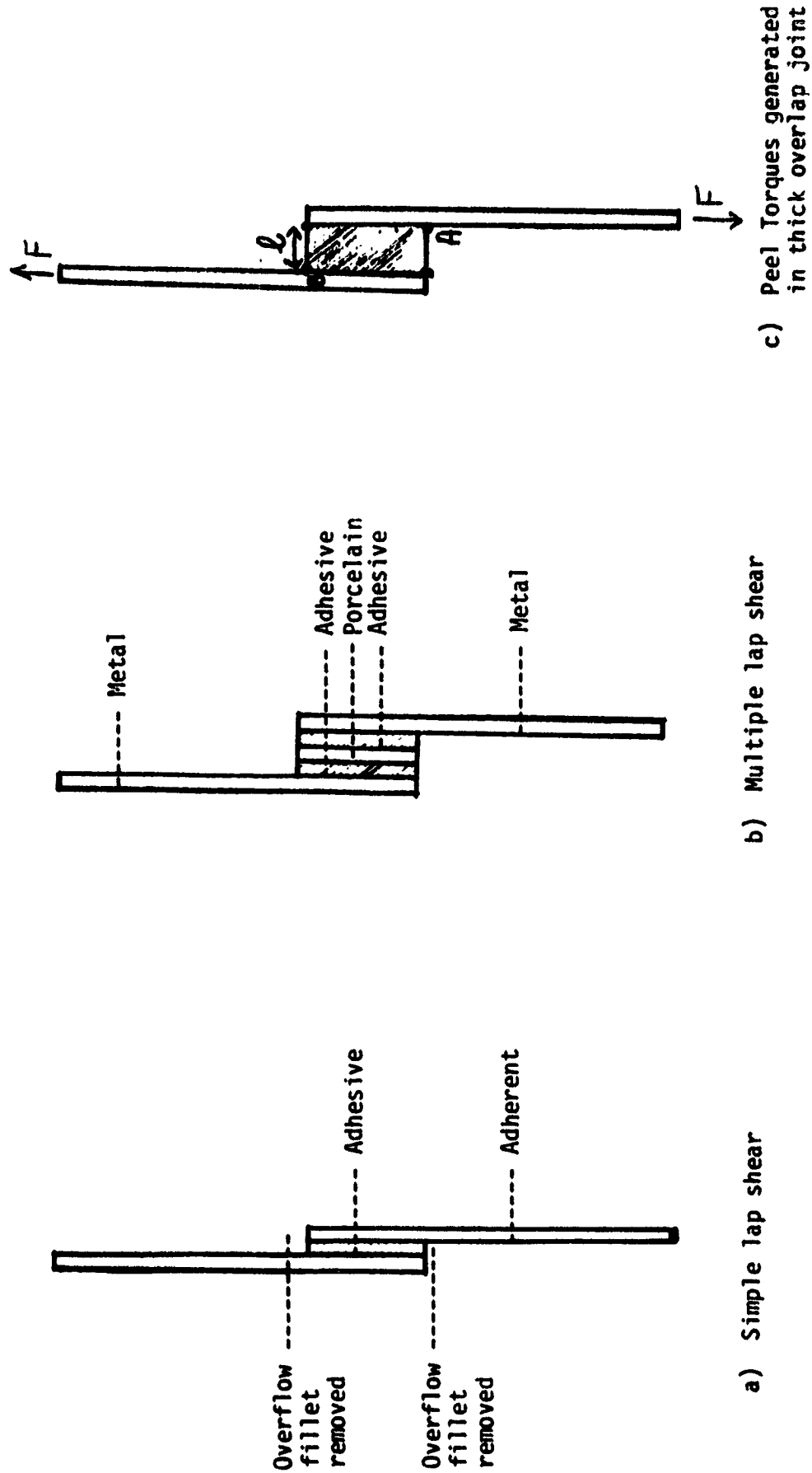
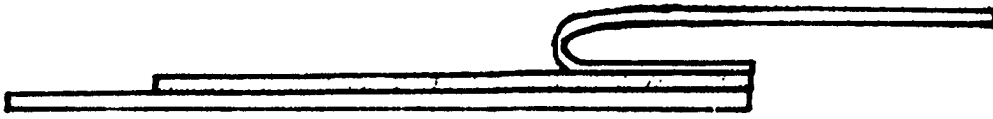
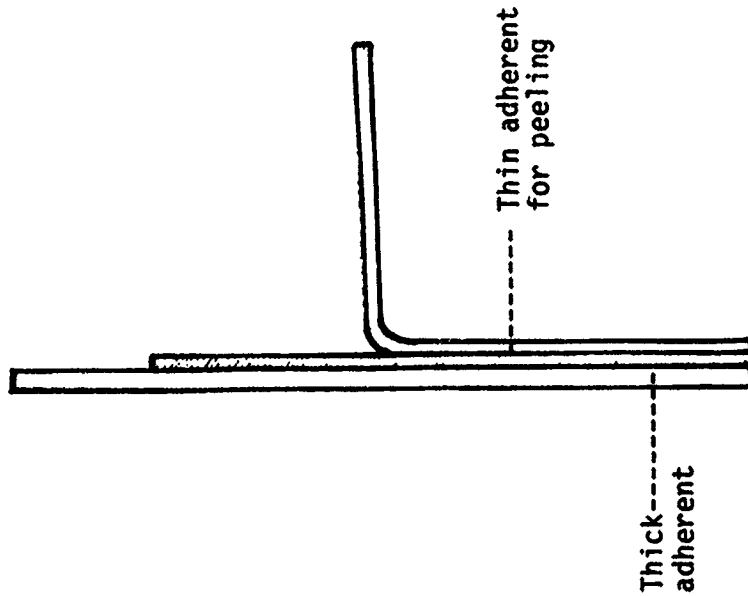


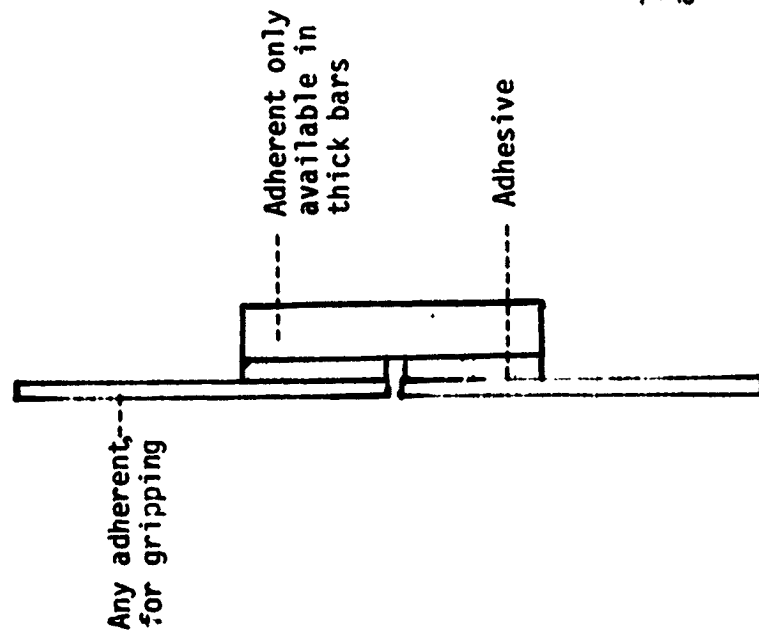
Figure 2: Adhesive Joints



f) Peel Sample



e) Peel Sample



d) Single Strap

Figure 2 Continued: Adhesive Joints

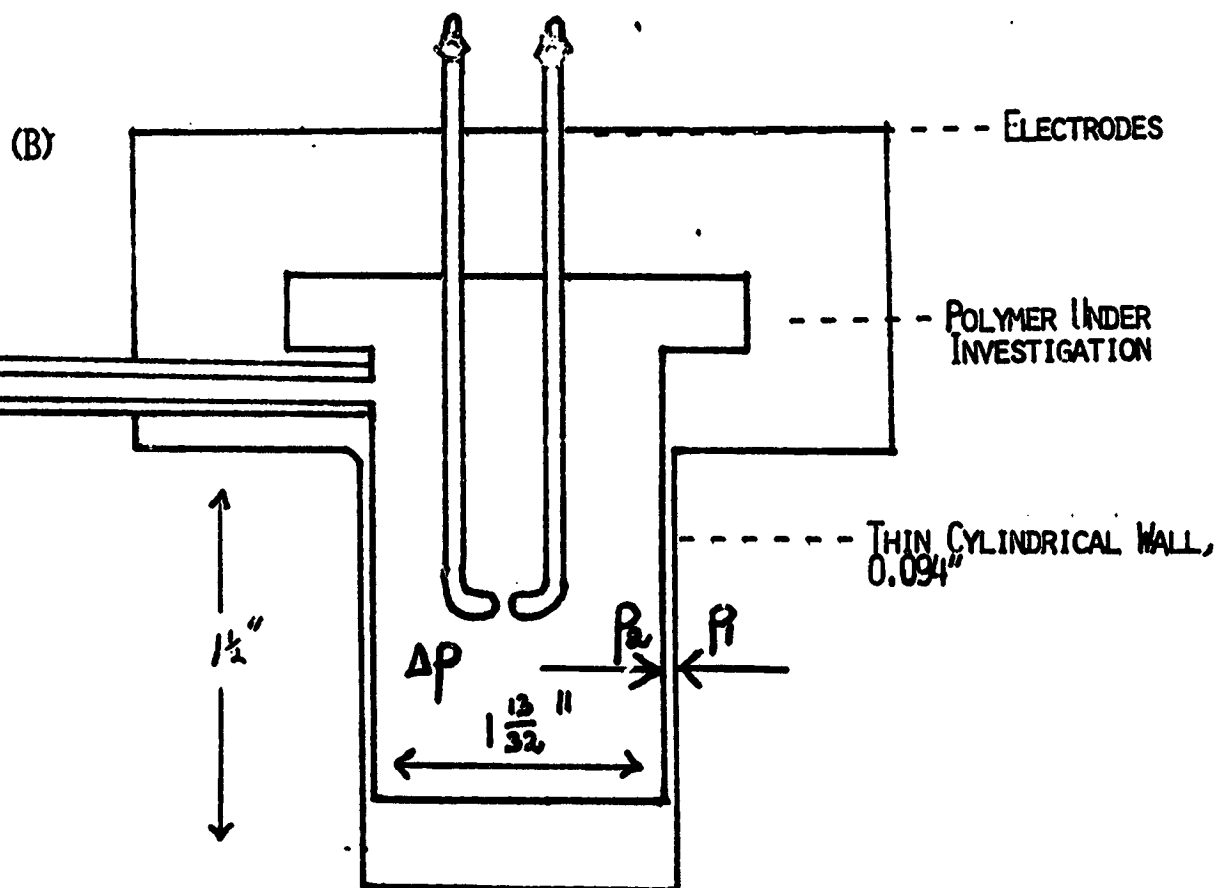
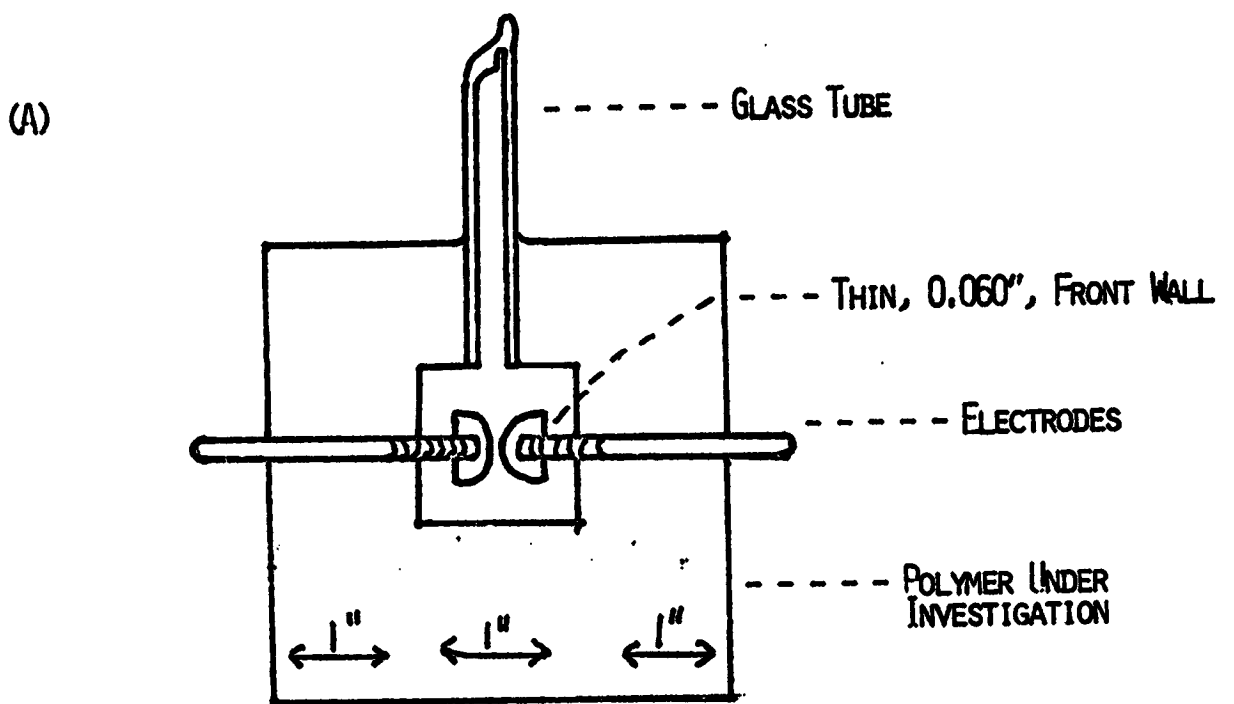
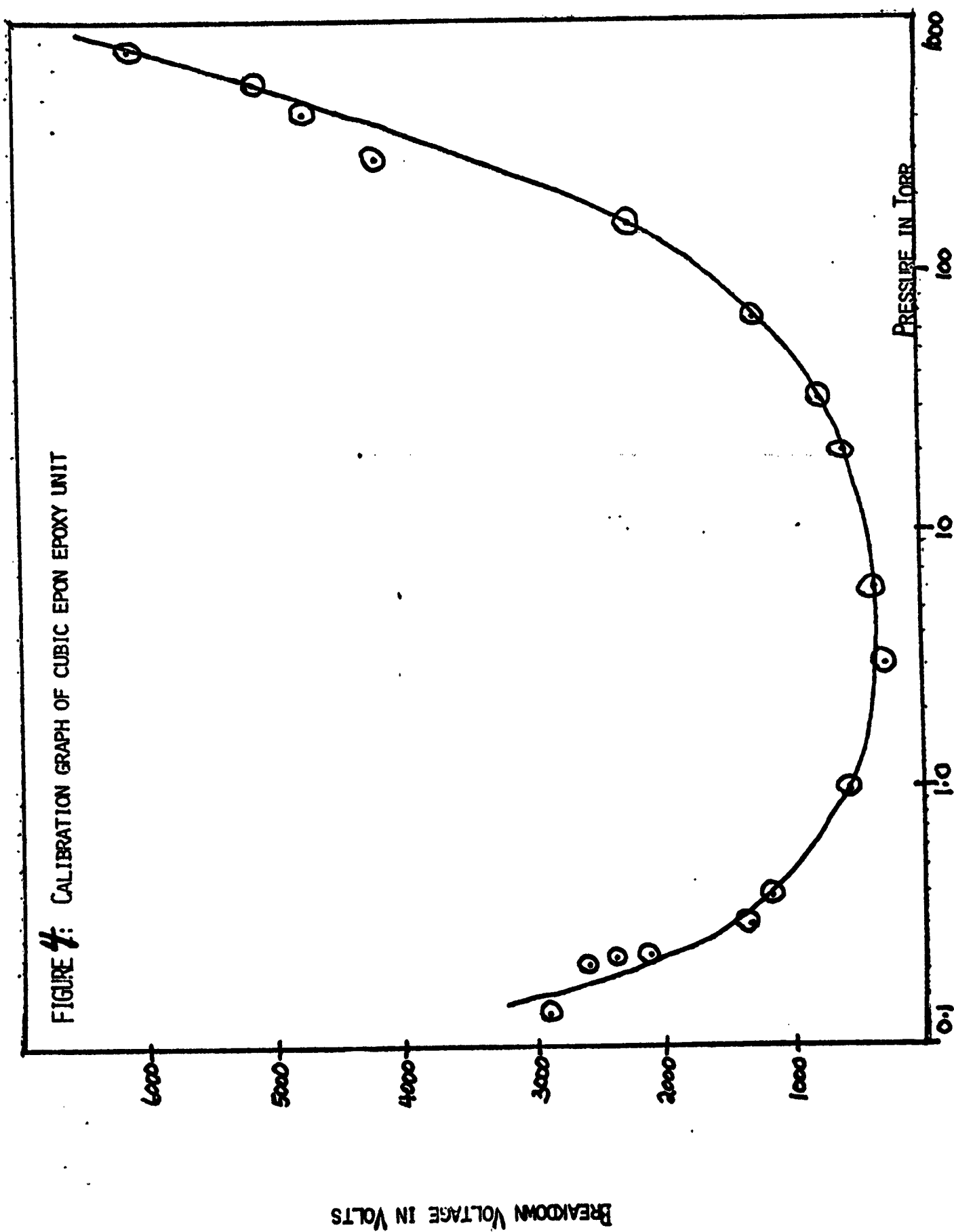


FIGURE 3: MOLDED (A) CUBIC, (B) CYLINDRIC UNITS

FIGURE 4: CALIBRATION GRAPH OF CUBIC EPON EPOXY UNIT



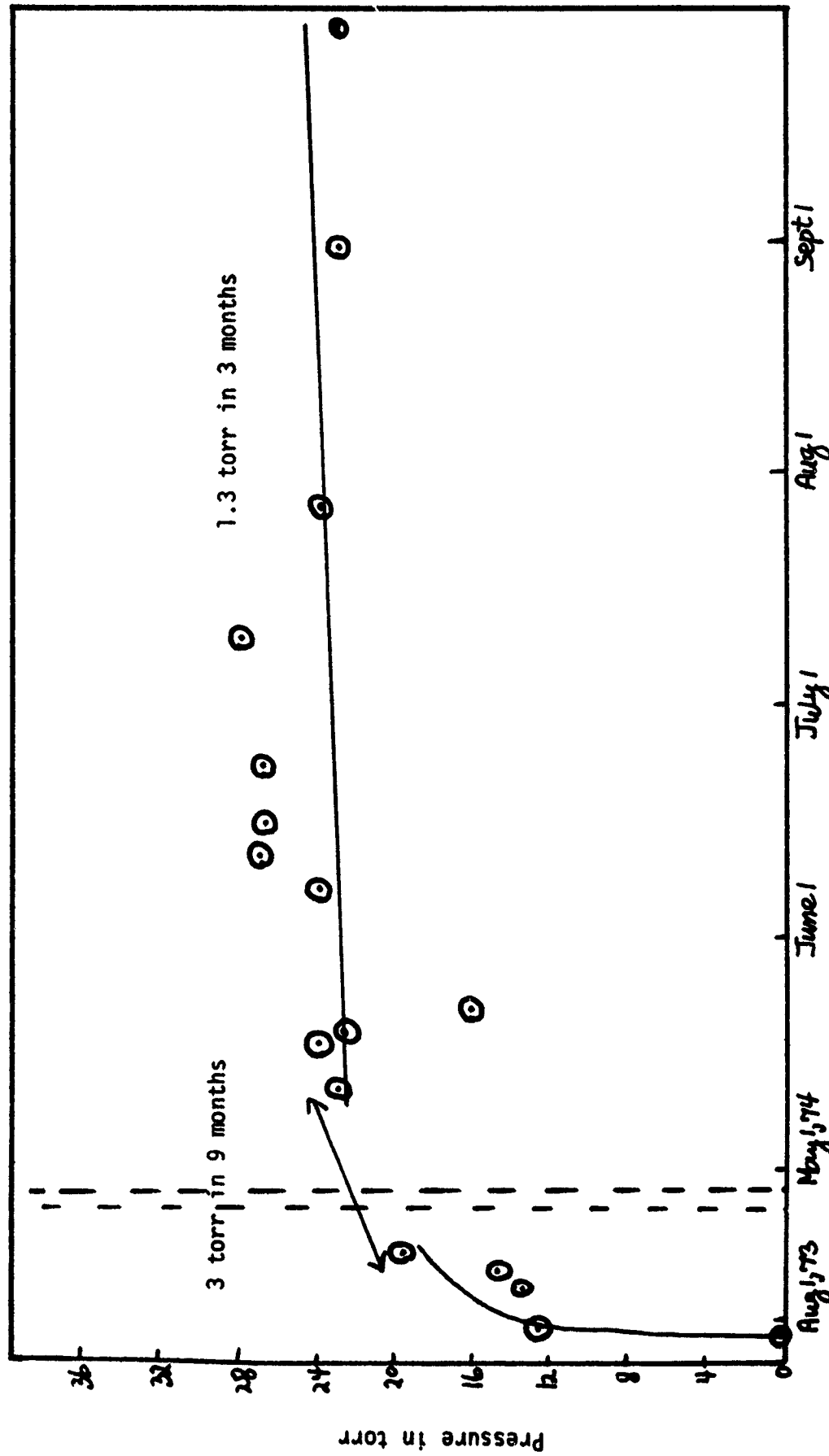


Figure 6: Pressure versus time for Stycast 3050, unit #2
(Sparkgap for pressure gauge)

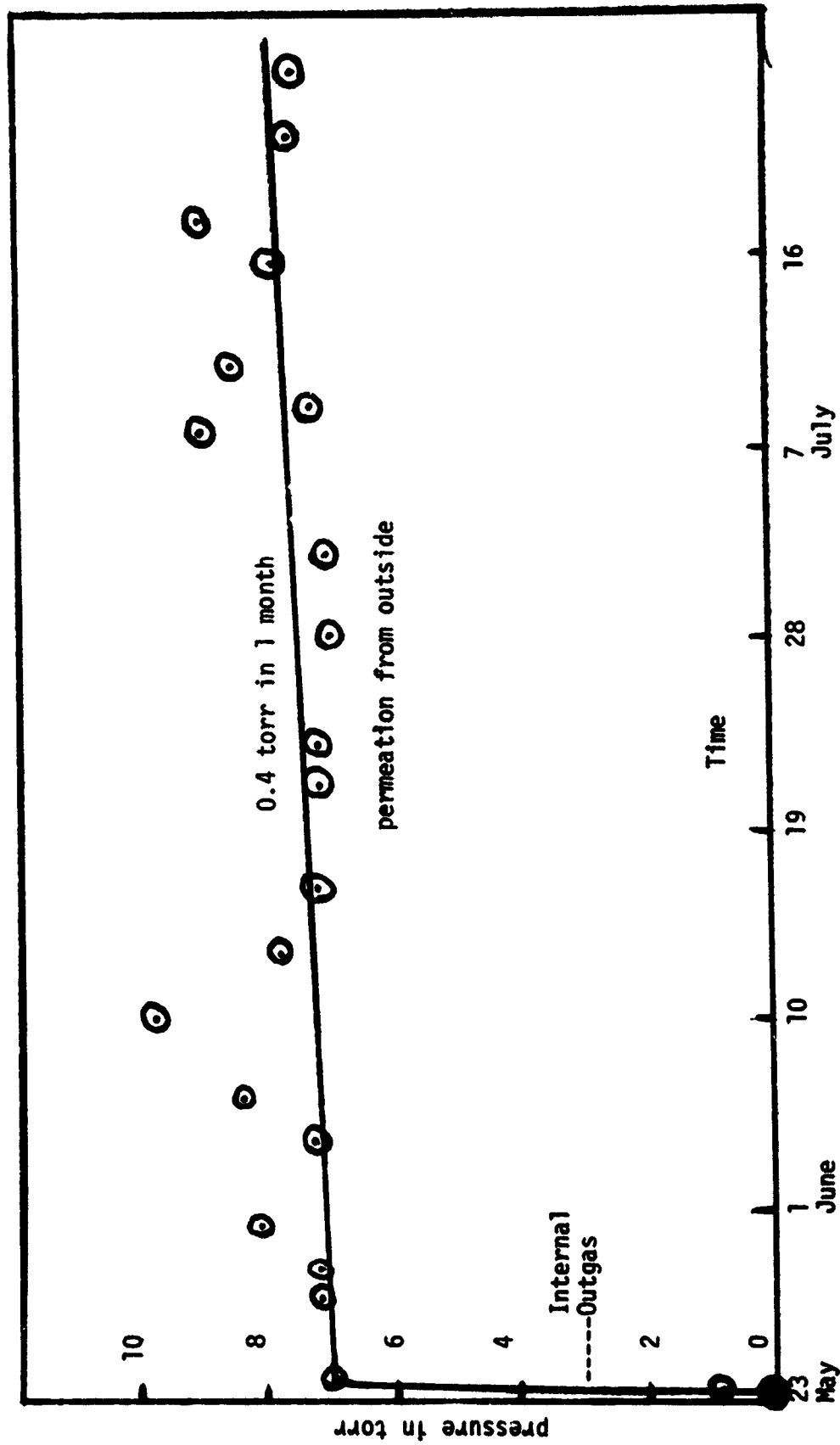
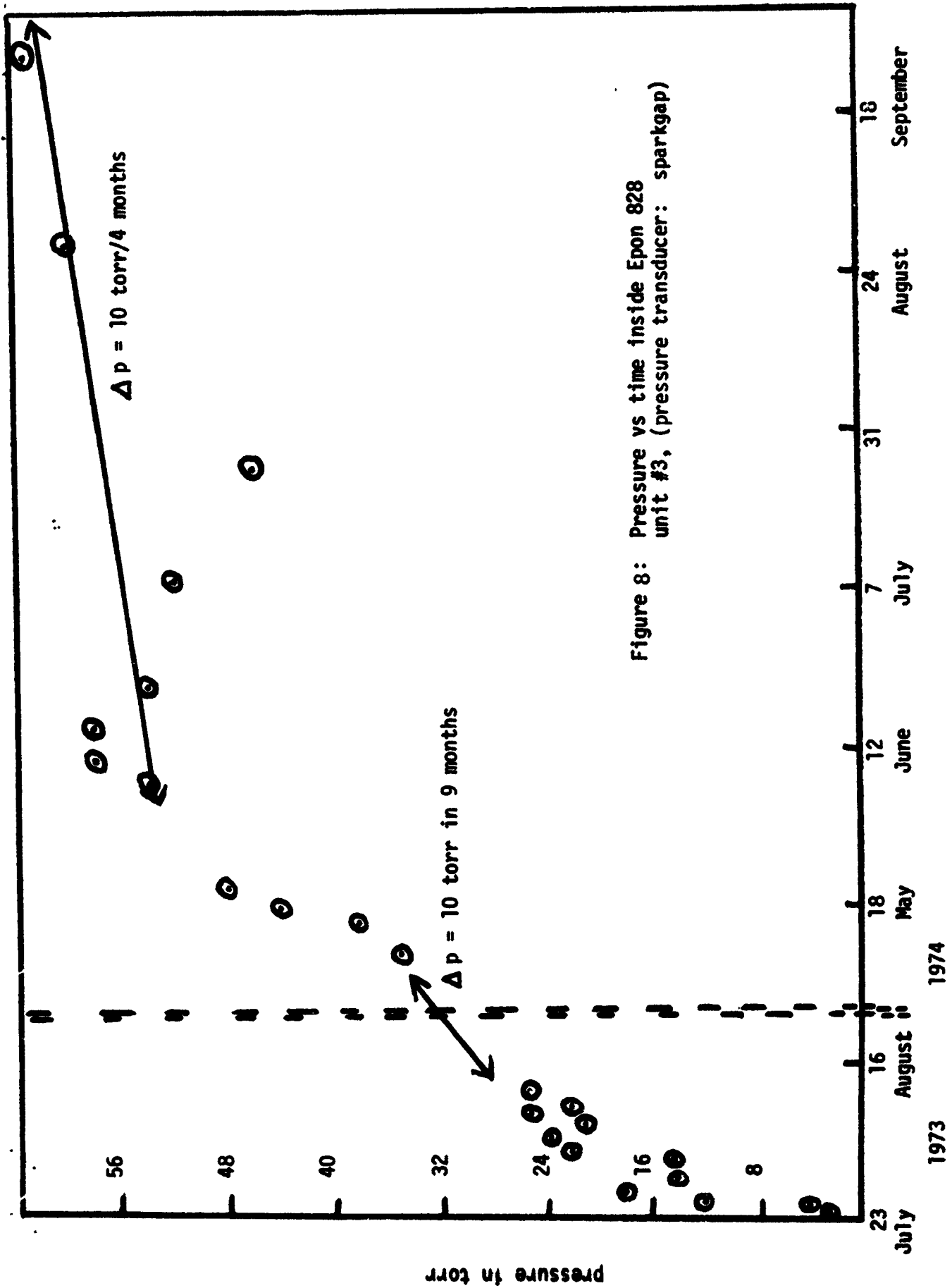


Figure 7: Pressure versus time for Stycast 3050 unit #3
(Pressure transducer: Thermocouple gage)



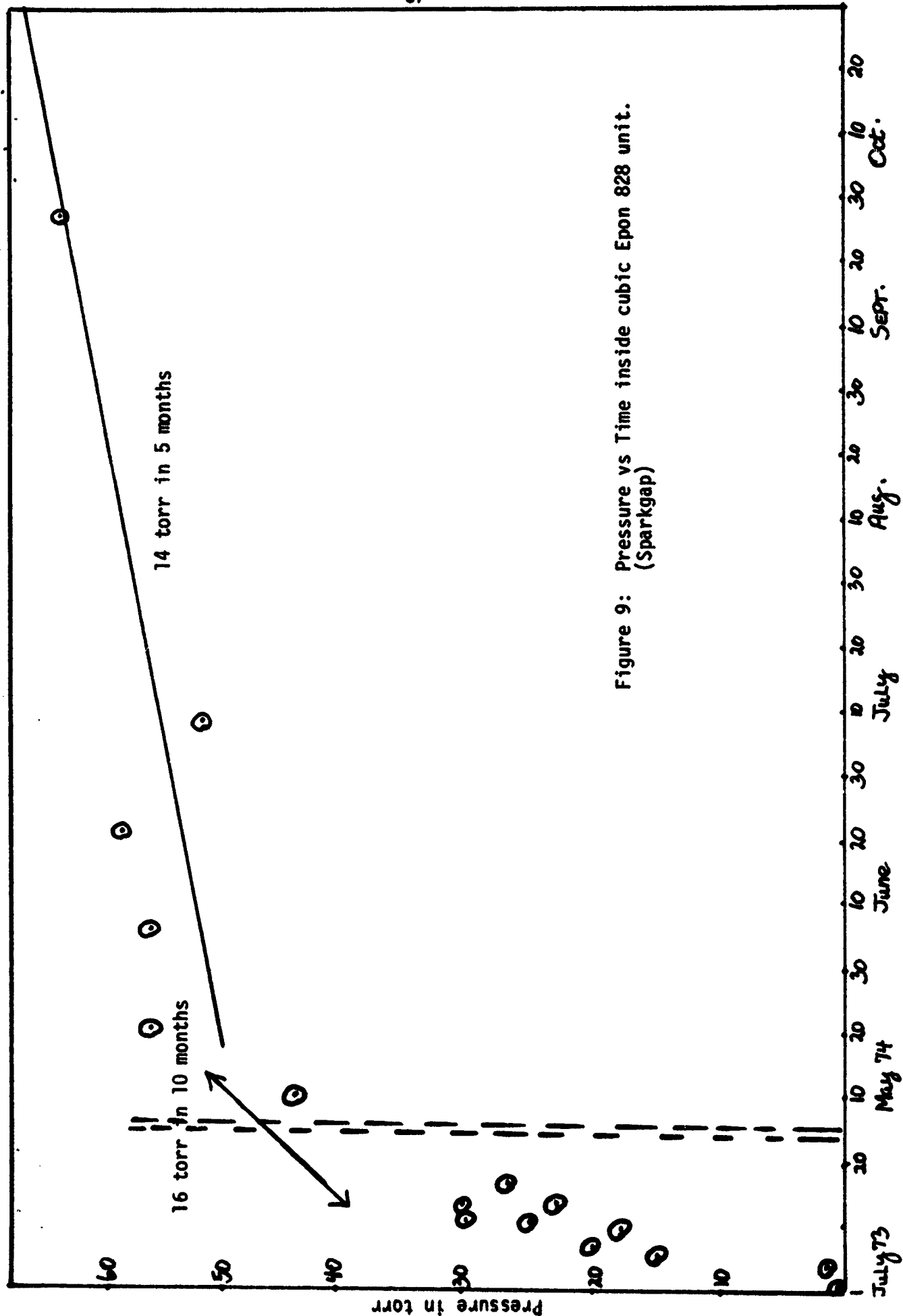


Figure 9: Pressure vs Time inside cubic Epon 828 unit.
(Sparkgap)

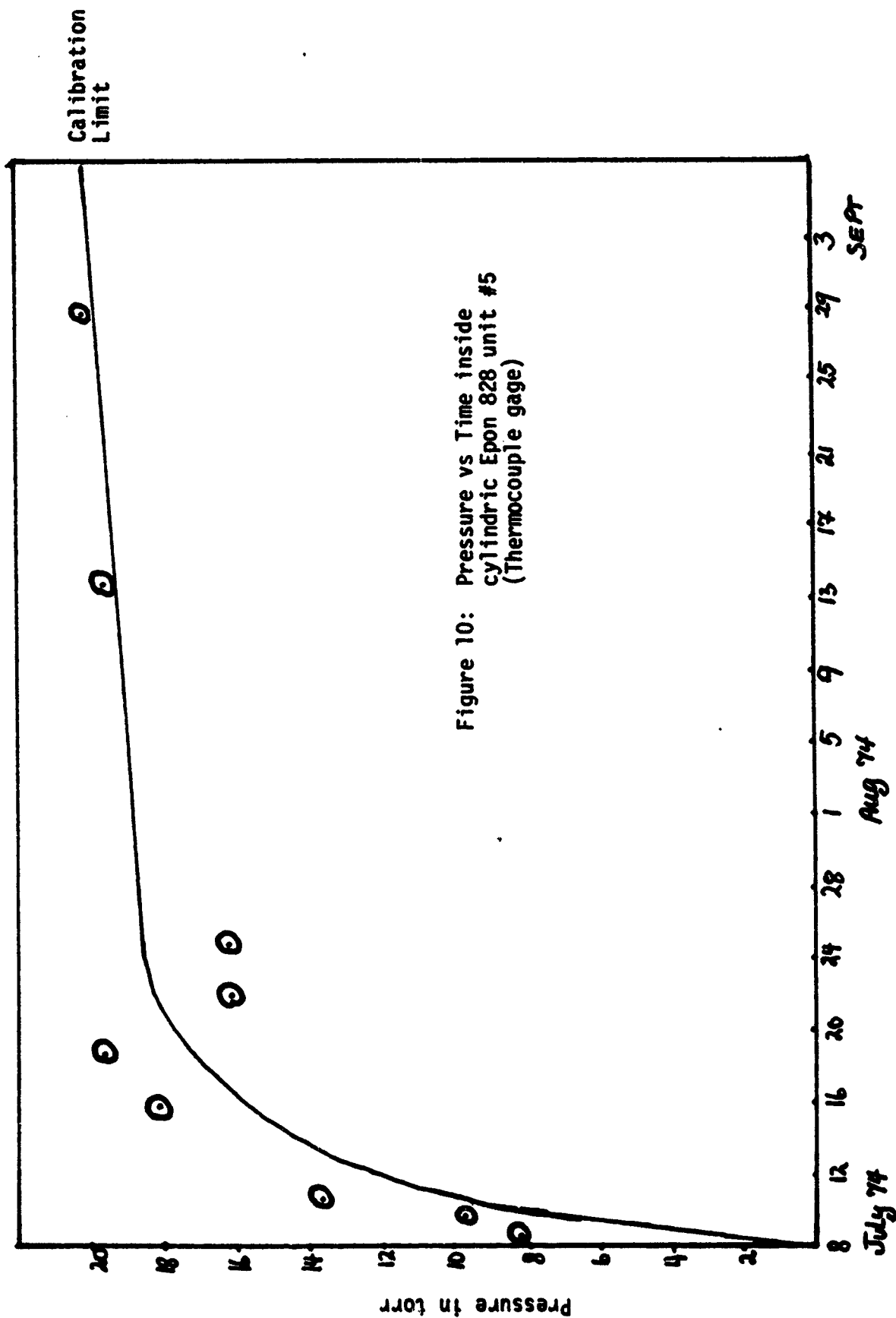
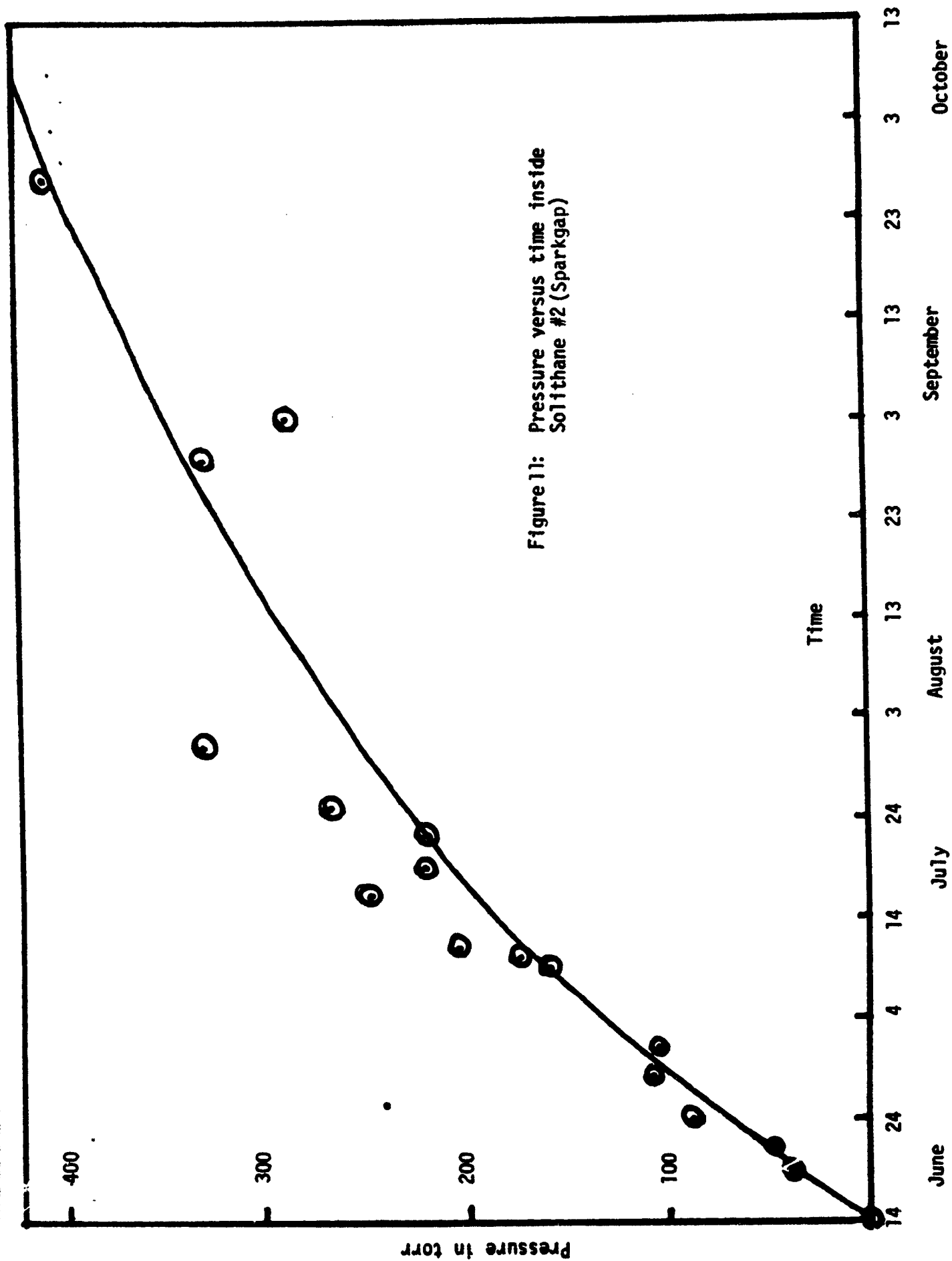
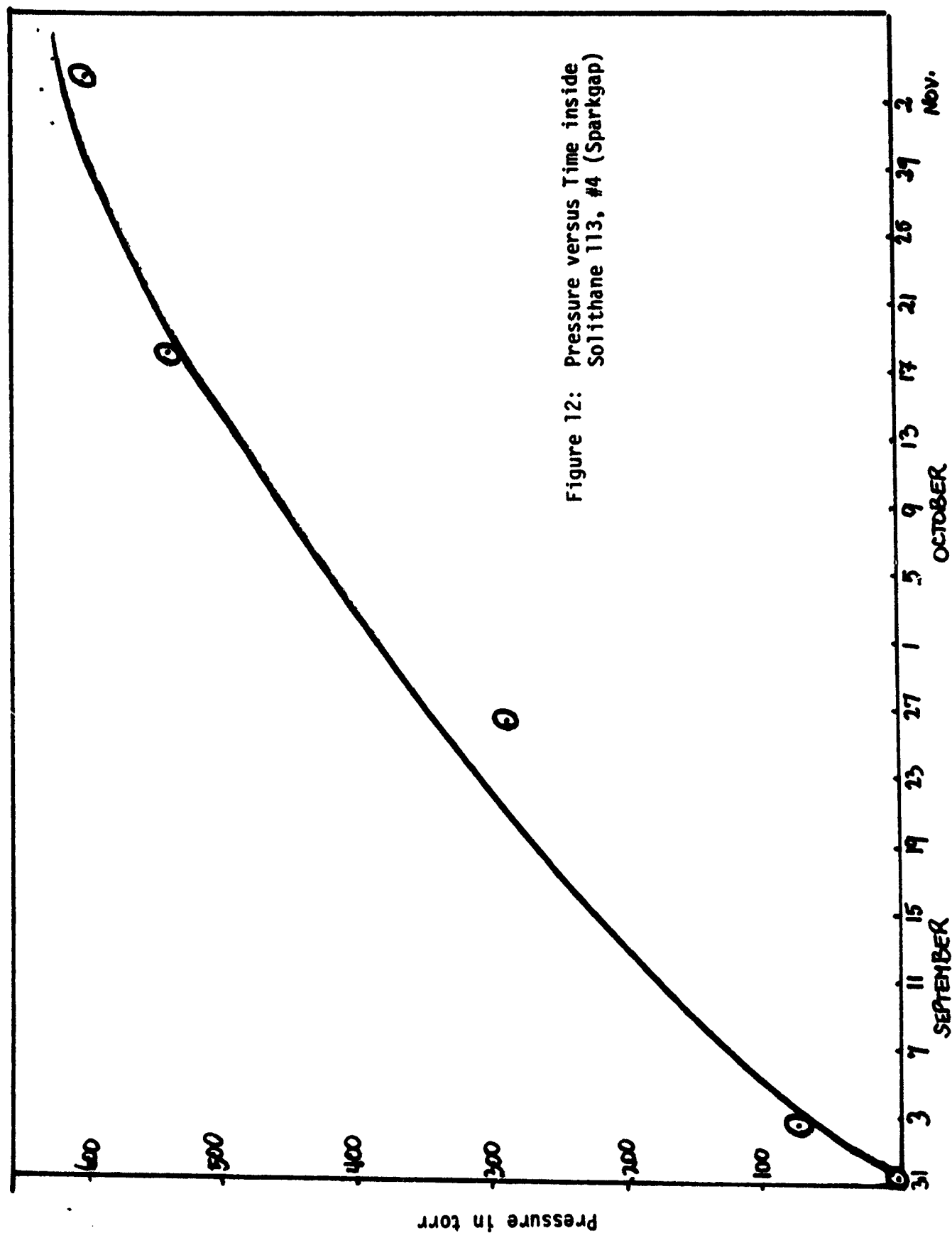


Figure 11: Pressure versus time inside Solithane #2 (Sparkgap)





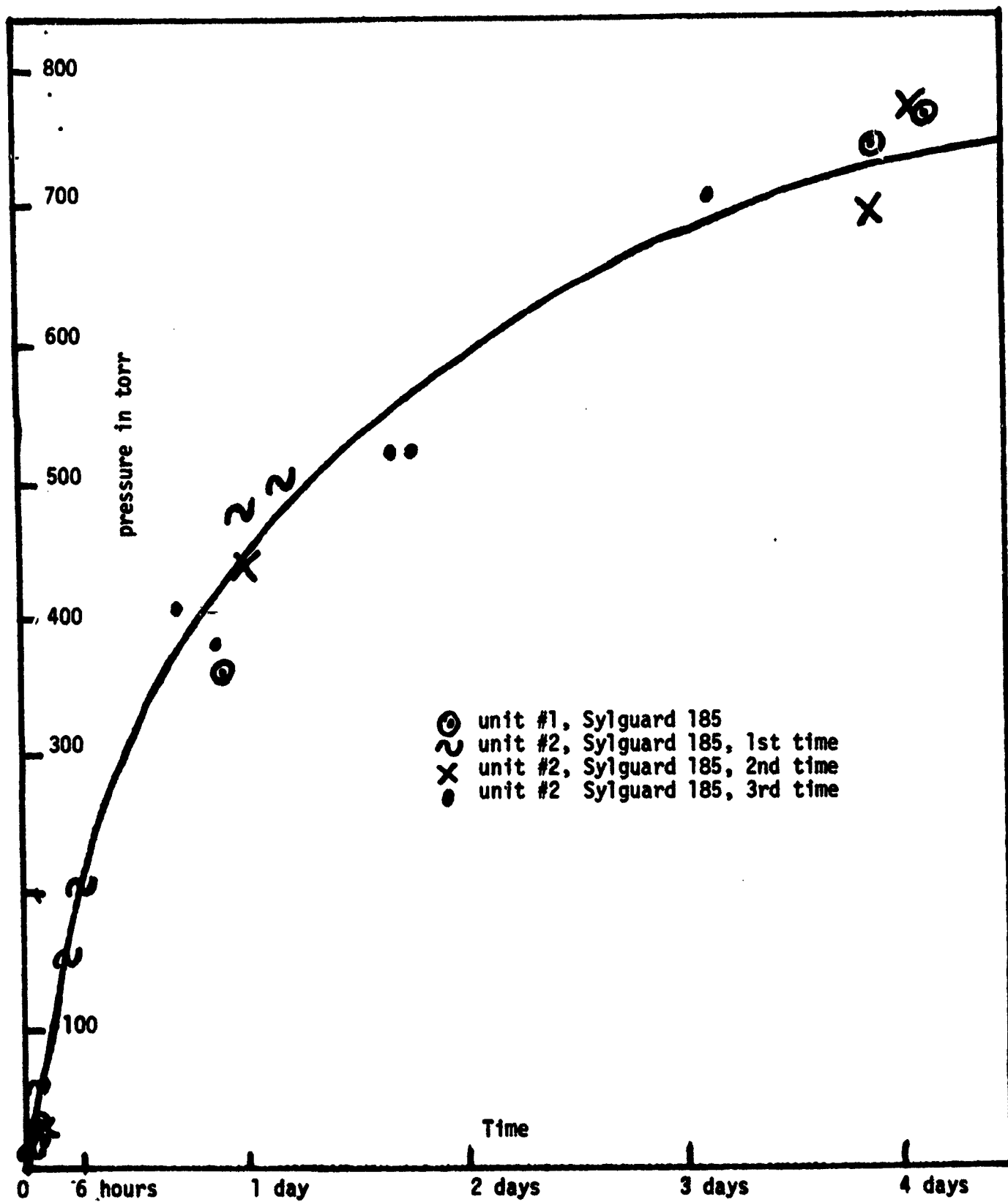
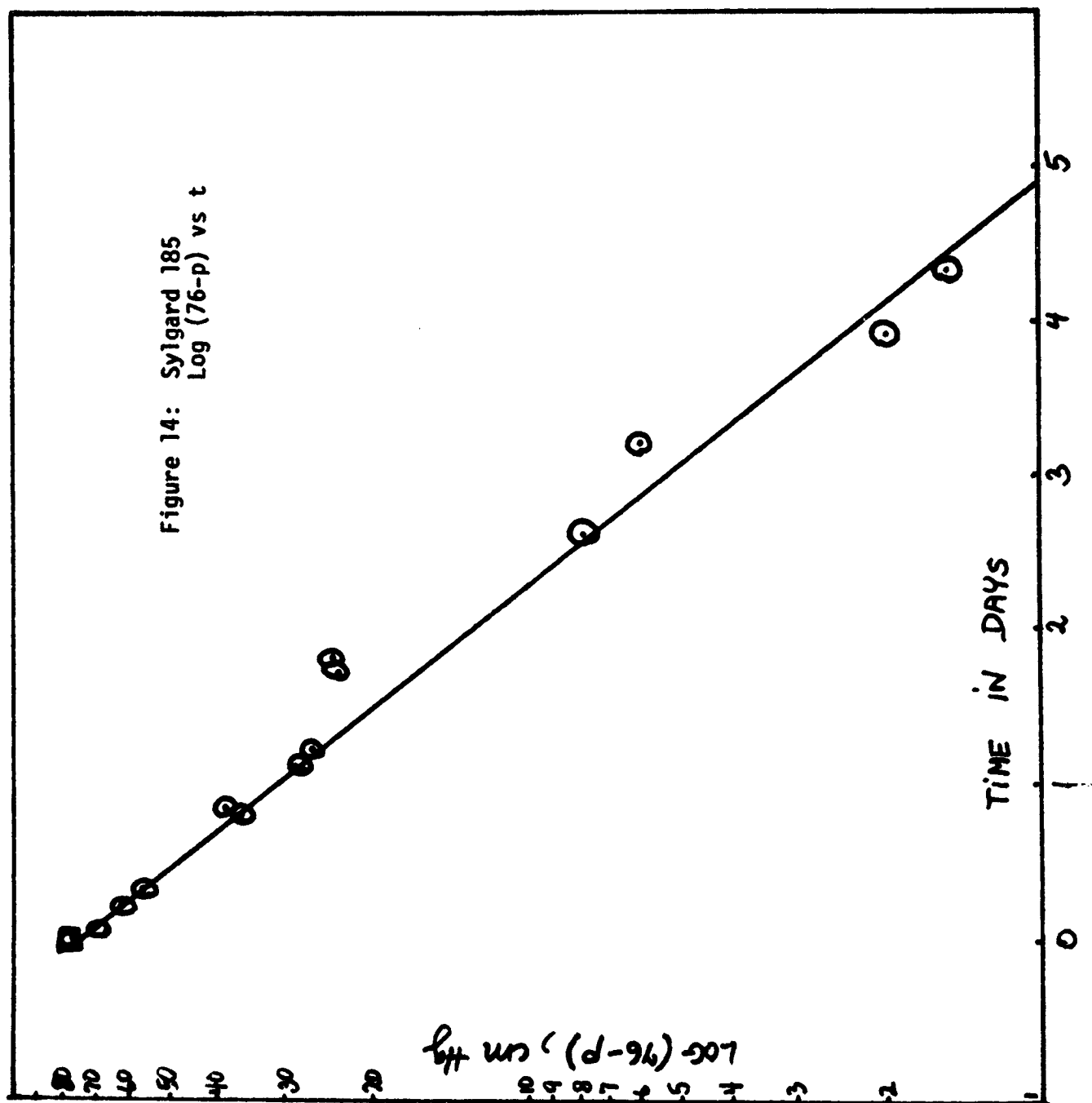
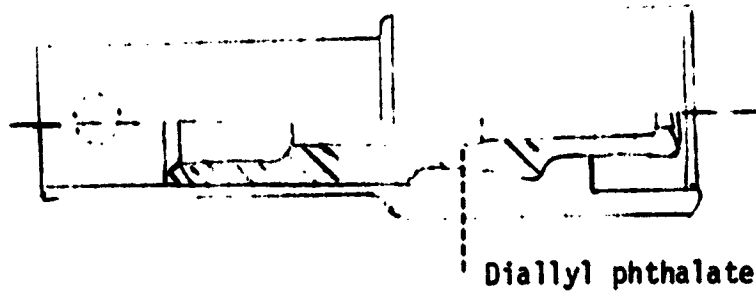
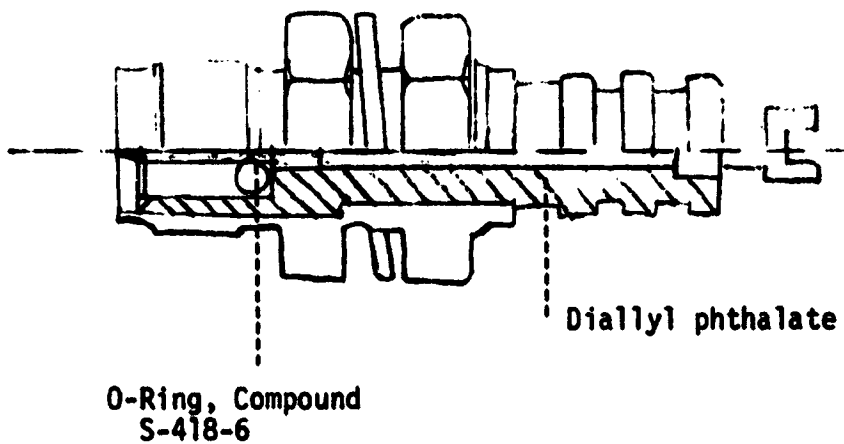


Figure 13: Pressure versus time for Sylguard 185





a) Female connector P/N 167-3770 A

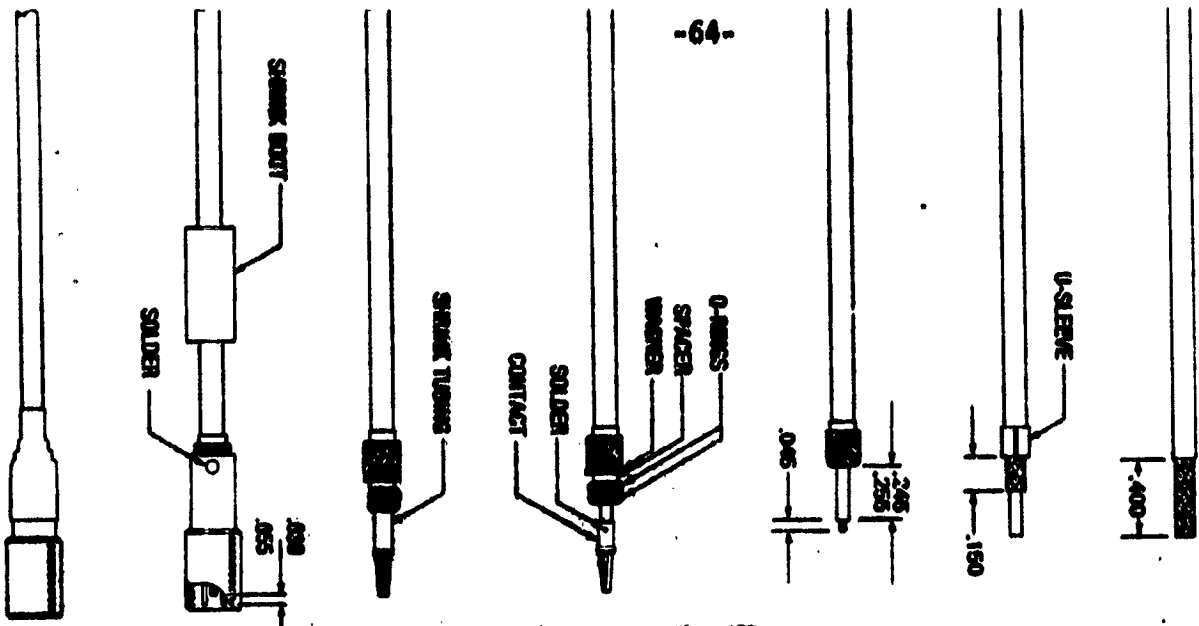


b) Male panel connector P/N 167-3771

Figure 15: Diagrams of Series 600 Reynolds connectors tested

INSTRUCTIONS PERTINENT TO P/N167-3770A ONLY.

Figure 16: Assembly of "L" cable with P/N 167-3770 A
Courtesy: Reynolds Industries



U-SLEEVE	SHRINK BOOT	WASHER	SPACER	O-RINGS	SHRINK TUBING	CONTACT	CONNECTOR BODY

SEALING INSTRUCTIONS

STEP 1
Cut cable to length, clean and square. Strip jacket to dimension, exposing braid. Note: if it is desired to have a moisture seal high altitude cable assembly, follow sealing procedures "A" through "G" on the right prior to proceeding to step 2.

STEP 2
Cut braid to dimension. Crimp U-sleeve into position as shown and turn back braid over U-sleeve as tightly as possible and tin dip. Remove all flux.

STEP 3
Strip inner insulation to dimension shown and then trim inner conductor to dimension and tin dip core. The inner conductor must have no severed strands and not more than two strands nicked during stripping.

STEP 4
Slide on washer and spacer. Lubricate two O-rings with Dow Corning FS1281 dielectric compound and slide on. Solder contact in place through hole per MIL-S-6872 using SN 60 solder per QQ-S-571. No gap allowed between end of contact and inner insulation and no solder buildup allowed on the contact outer diameter. Clean thoroughly.

STEP 5
Shrink tubing in place at 300° to 320° F and allow to cool undisturbed to prevent formation of a cold solder joint.

STEP 6
Slip boot over cable. Insert assembled cable into body coupling nut assembly until the contact shoulder bottoms out in the insulator. Solder body in position using a fixture to maintain pressure on the cable in the body. Solder through two holes per MIL-S-6872 using SN 60 solder per QQ-A-571. Clean thoroughly.

STEP 7
Shrink boot into position as shown at 300° to 320° F.

STEP A
Immerse both ends of the cable length in a container of silicone resin adhesive to a depth of $2\frac{1}{2}'' \pm \frac{1}{4}''$. If it is desired to seal only one end of cable it is necessary to cut cable to twice the length of the required finished cable length so that both ends can be immersed in the sealant.

STEP B
Place the container with cable in a suitable vacuum chamber and evacuate the chamber gradually until a pressure of 10 mm HG absolute is obtained. NOTE: The solvent in the adhesive system is quite volatile. In order to prevent overflow due to foaming and to minimize thickening of the solution, the vacuum should be applied gradually and intermittently and the pressure should not be allowed to drop much below 10 mm HG.

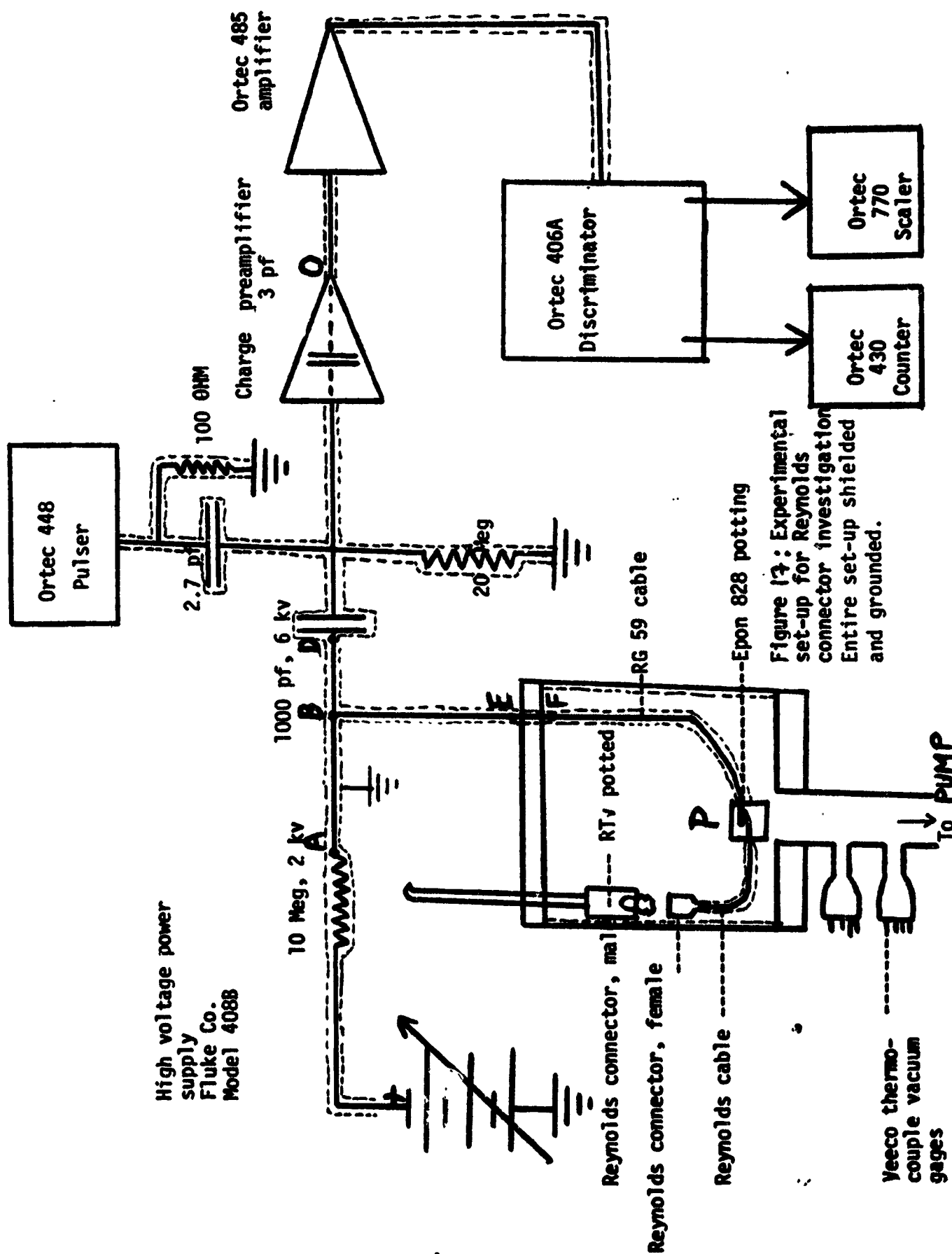
STEP C
Turn off vacuum pump, but maintain pressure at approximately 10-12 mm HG for 10-15 minutes to permit air to be evacuated from interstices of the cable.

STEP D
Break vacuum and restore atmospheric pressure, but keep the cables immersed in the resin for at least 2 hours to allow resin to flow into cable interstices.

STEP E
Remove cable from resin and wipe off excess resin using a clean dry cloth or paper-wipe.

STEP F
Using a clean lint-free cloth or paper-wipe moistened with Methyl-ethyl-ketone (MEK), complete the cleaning operation by rolling the adhesive off the cable ends.

STEP G
Place cleaned cable lengths in a forced draft oven set at 160-200° F and allow to dry for at least 2 hours. Proceed to step 2 on the left.



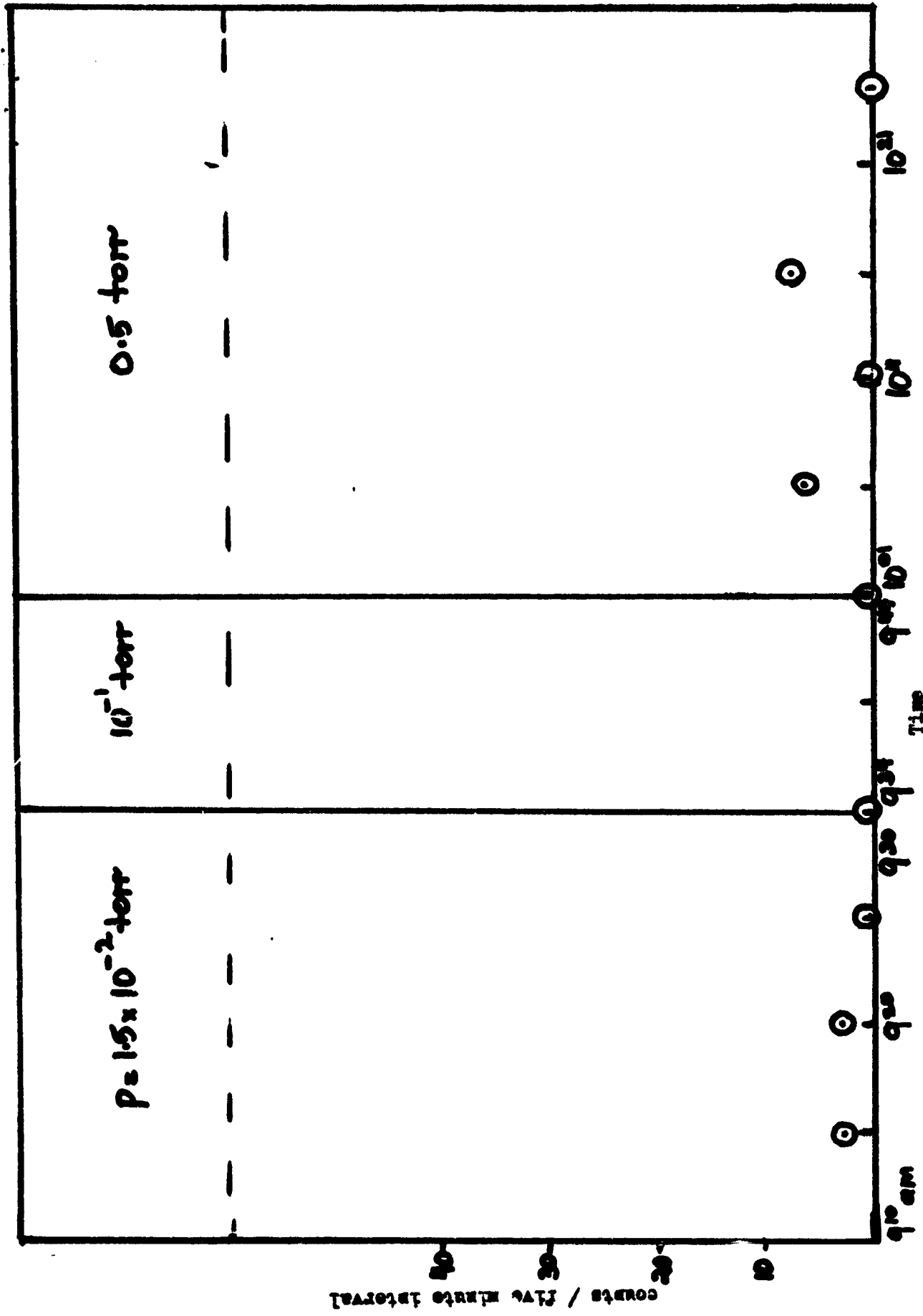


Figure 16a: August 15, 74. Series 600 Reynolds counter, filled at different pressures with ordinary air.

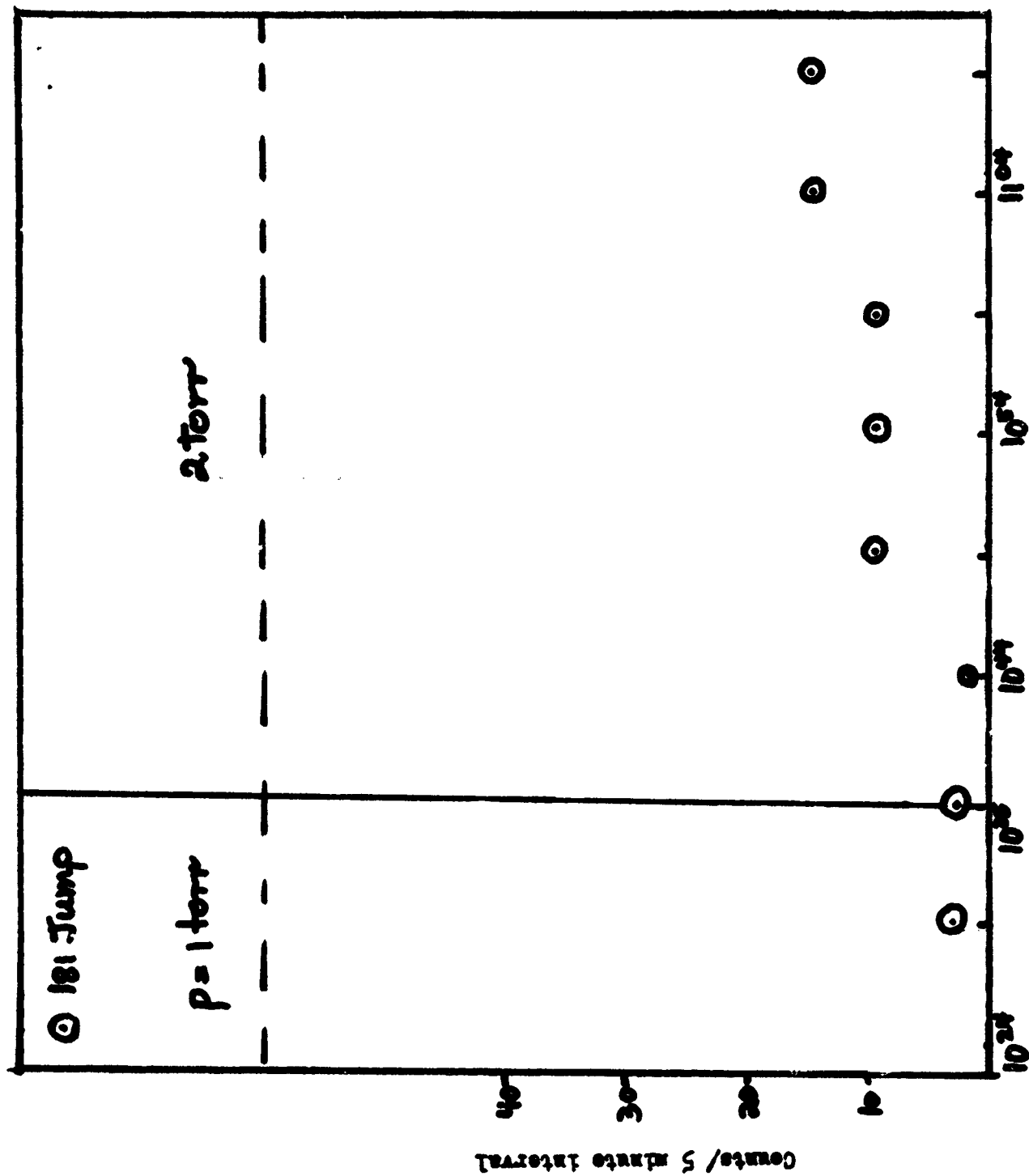


Figure 16b: August 15, 74: Series 600 Reynolds connector, filled at different pressures with ordinary air

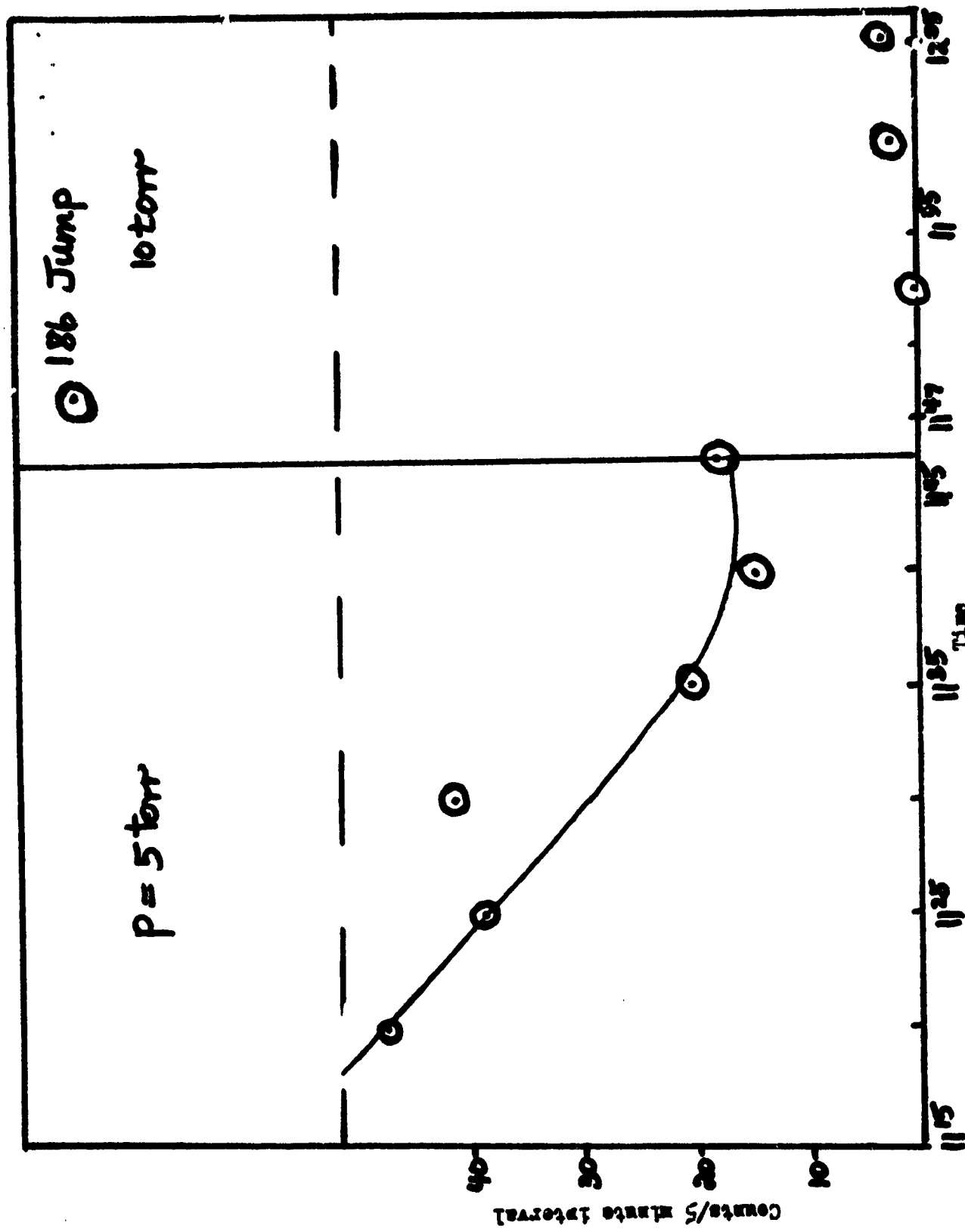


Figure 186: August 15, 74: Series 600 Reynolds connector, filled at different pressures with ordinary air

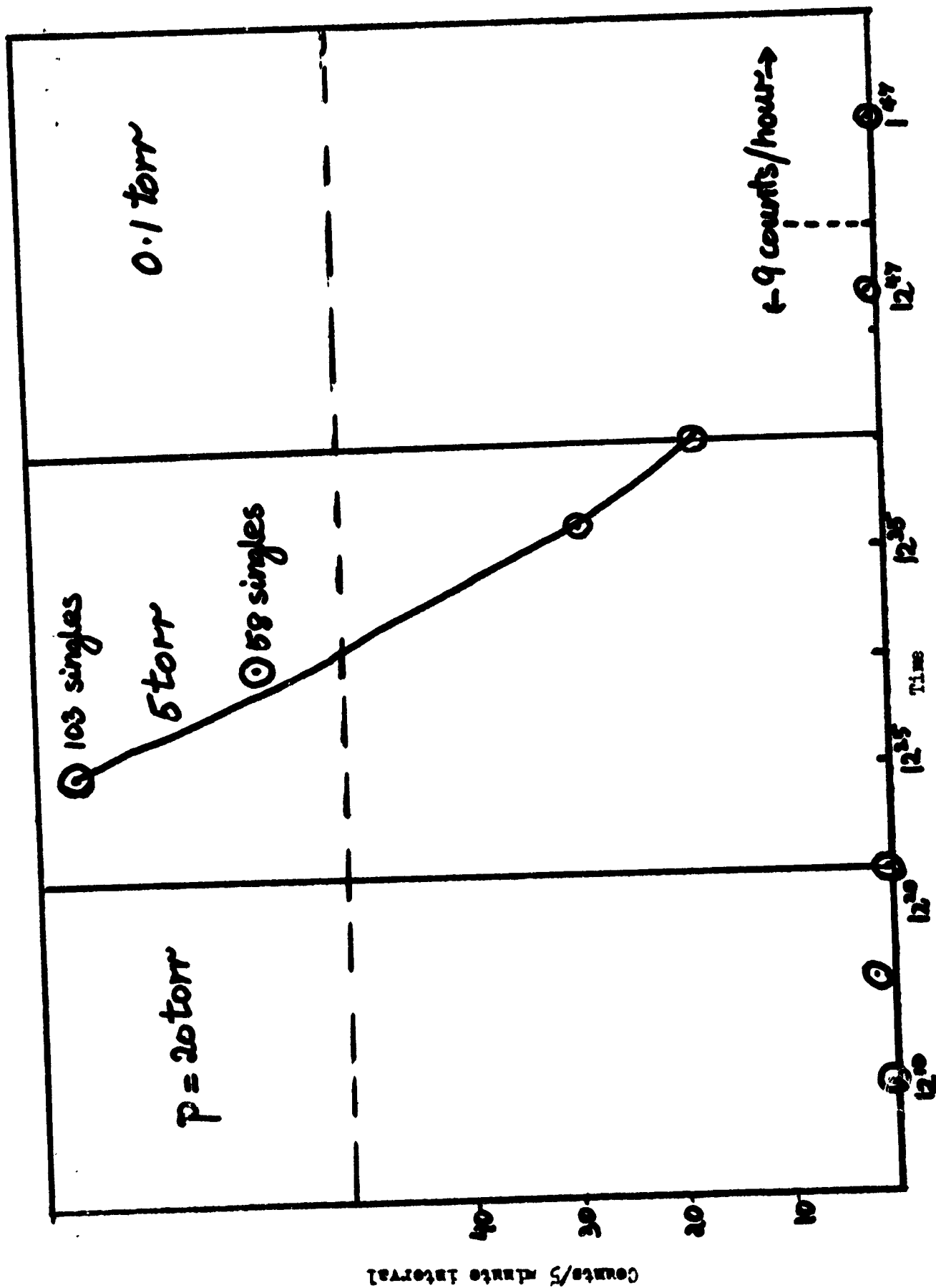


Figure 101: August 15, 74: Series 600 Reynolds connector; filled at different pressures with ordinary air.

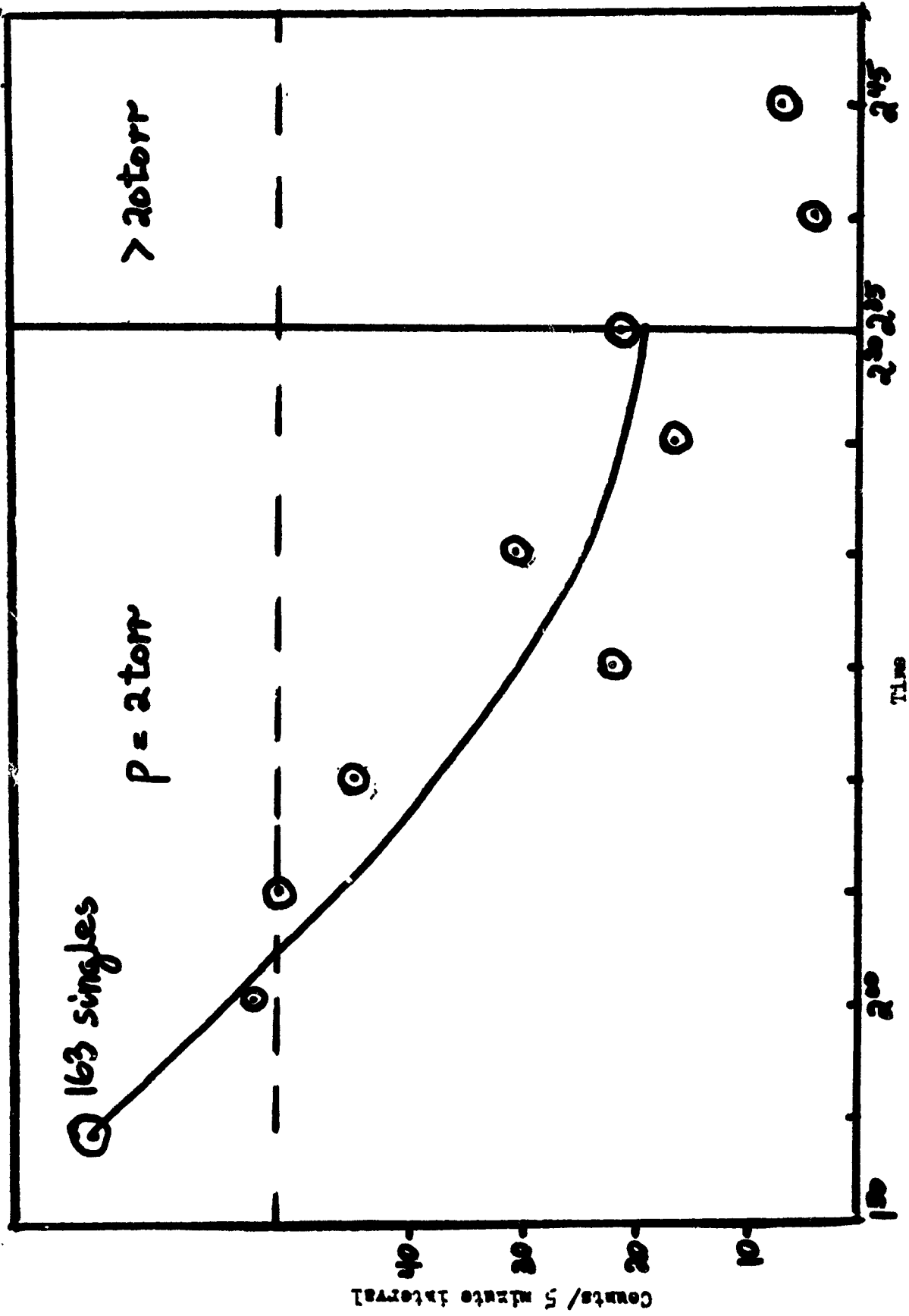


Figure 10e: Series 600 Reynolds cornet, filled at different pressures with ordinary air, August 15, 71

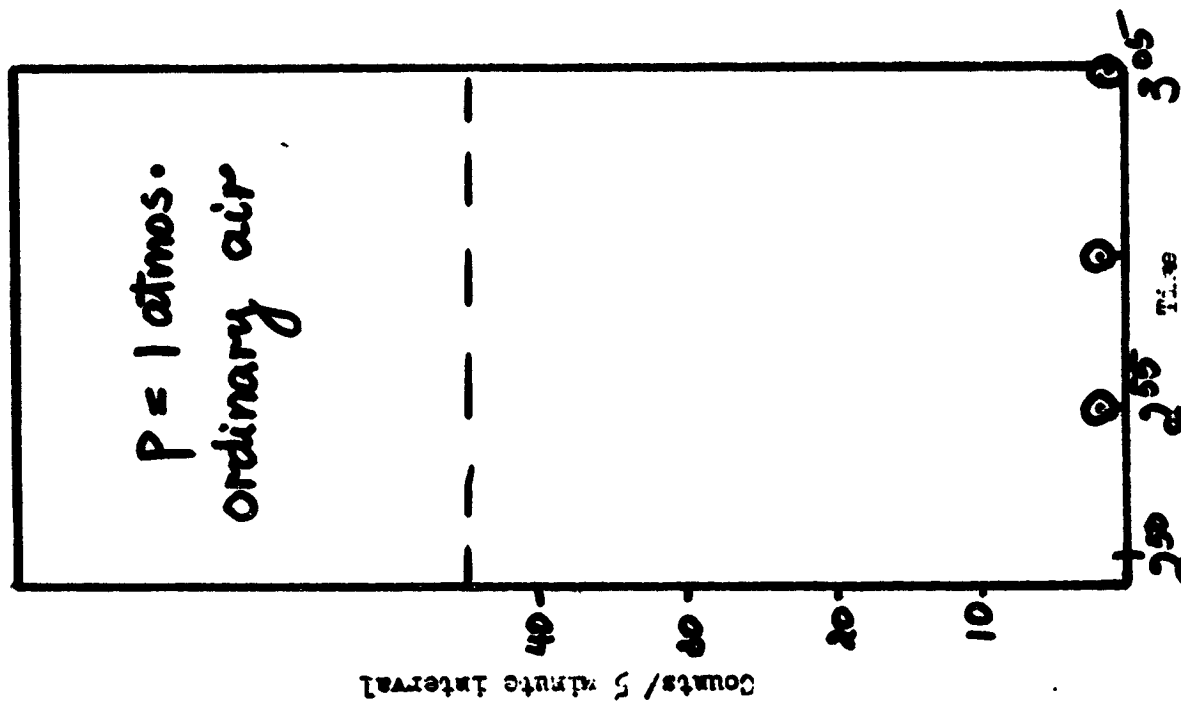


Figure 18: August 15, 74: Series 600 Reynolds connector, filled at different pressures with ordinary air

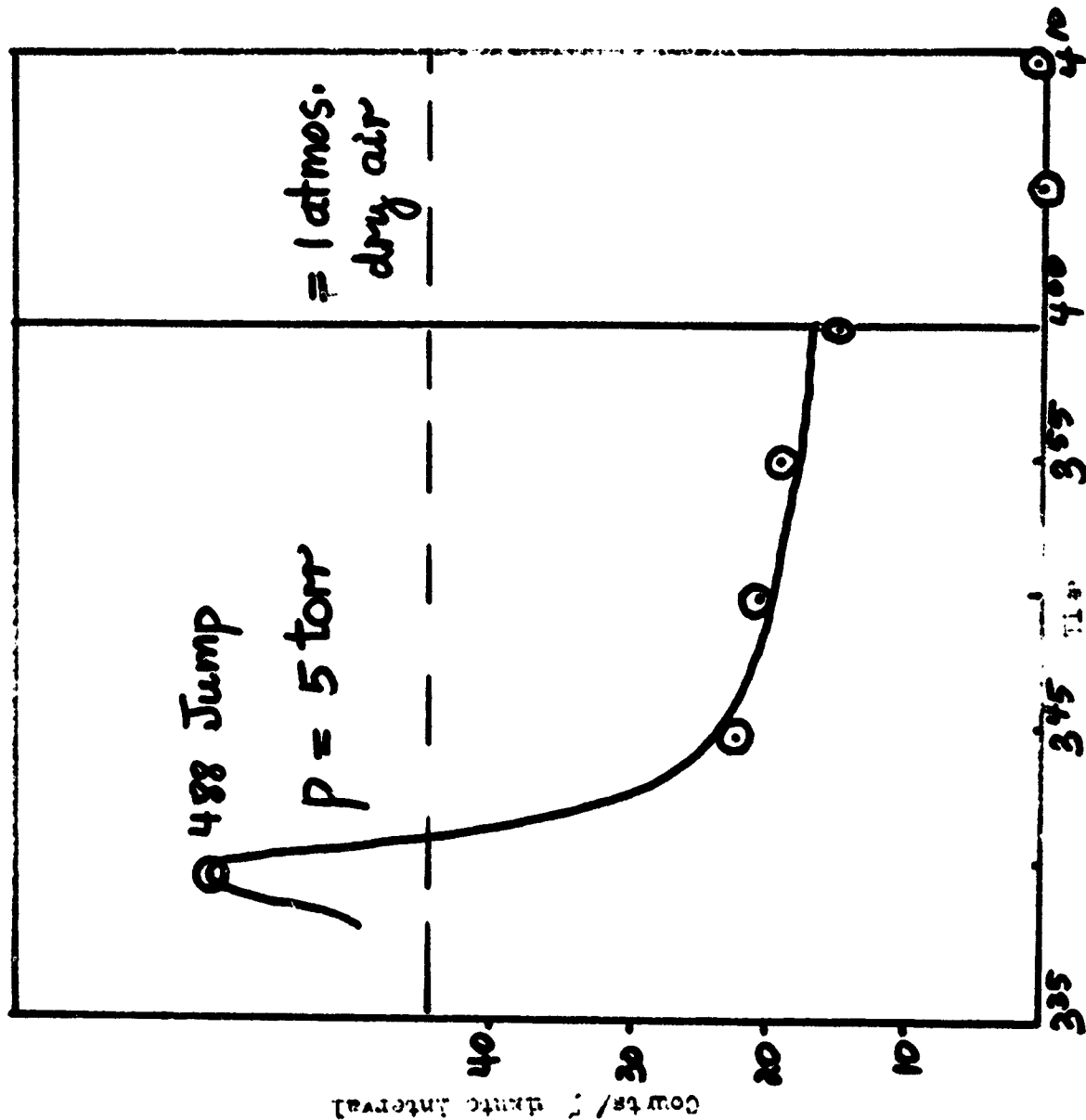


Figure 19: Series 600 Regol's connector, August 15, 74, filled at different pressures with constant dried air

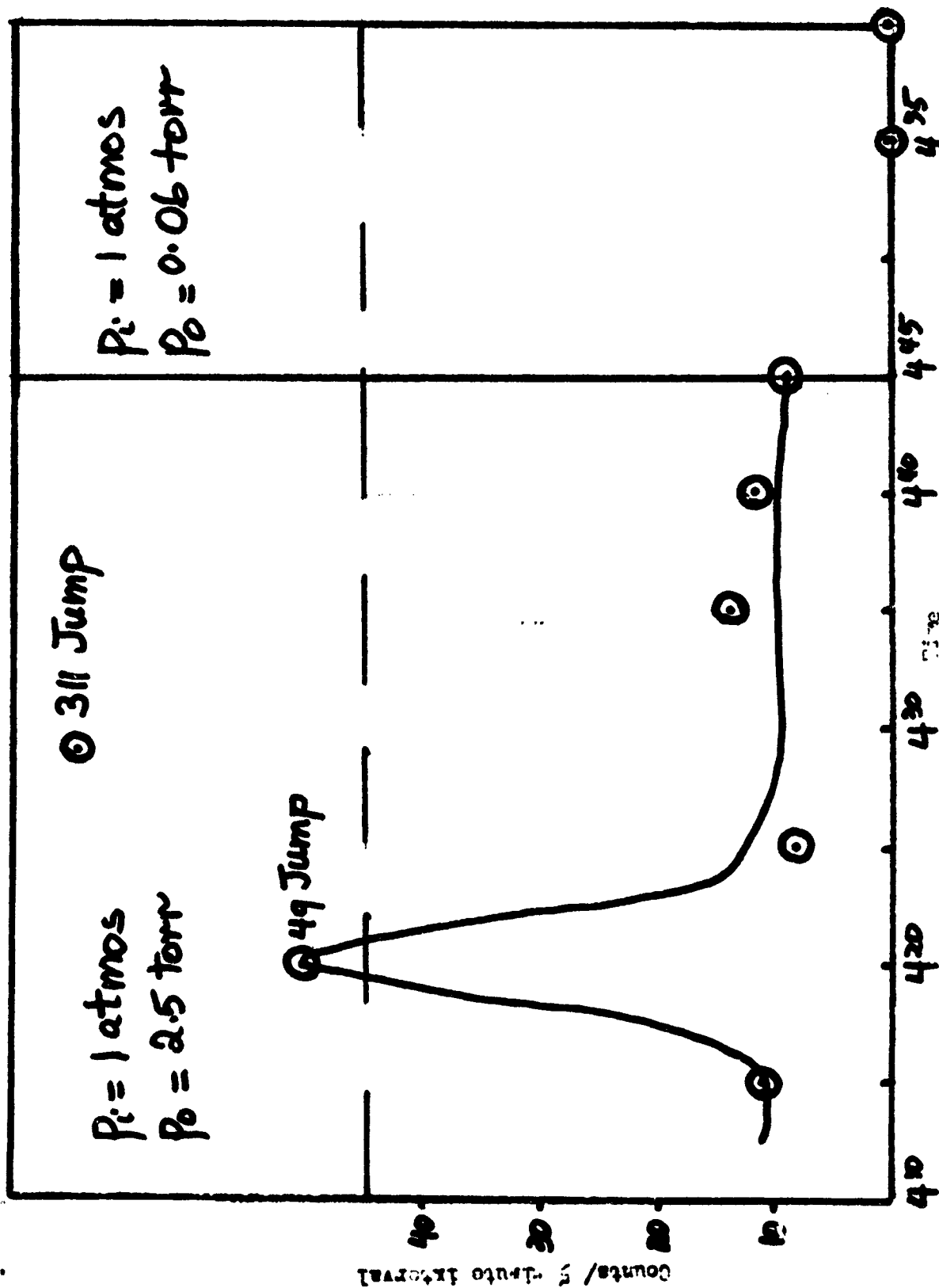


Figure 17b: August 15, 74: Series 600 Reynolds corneator, filled at different pressures with desiccant dried air

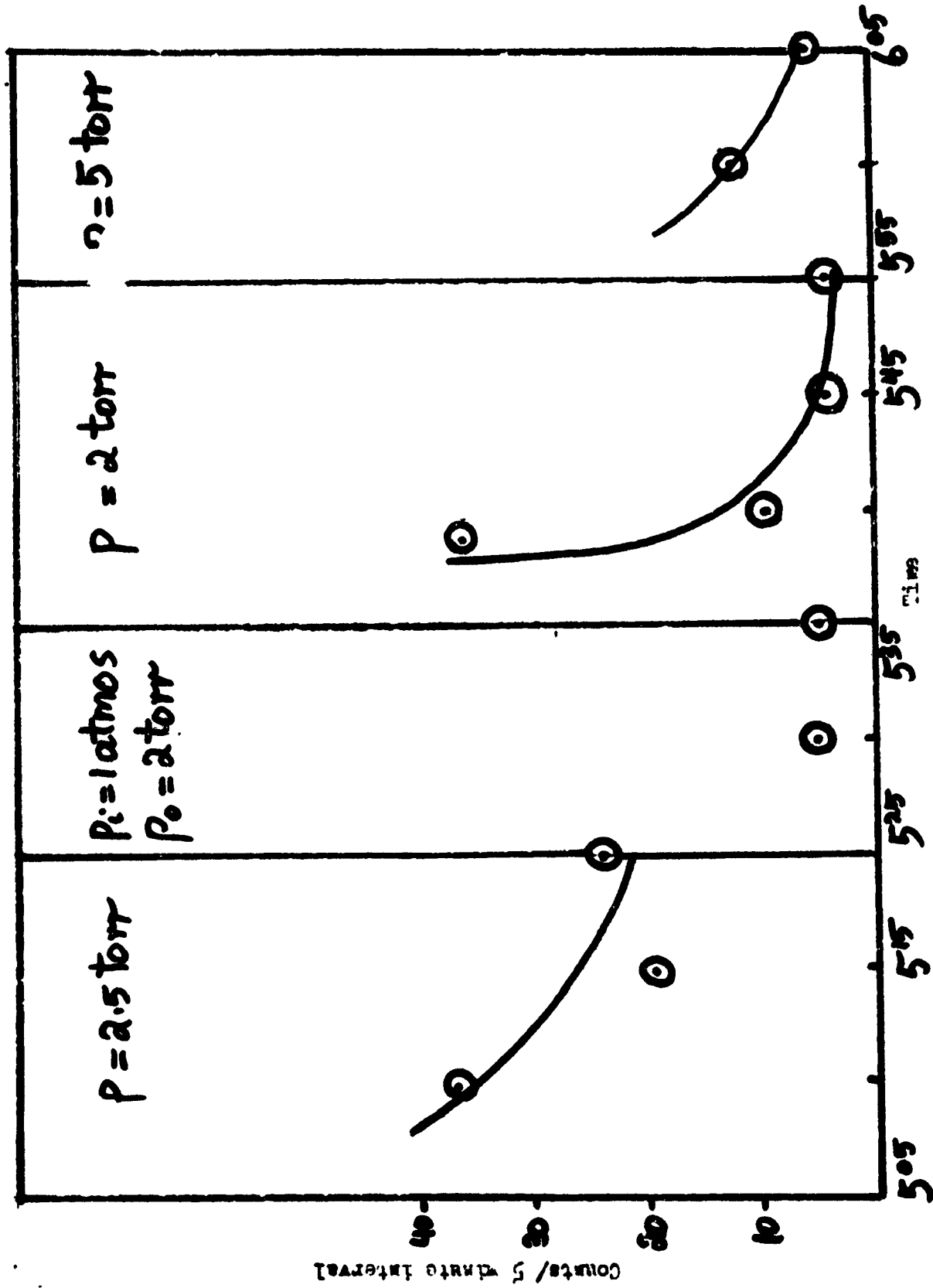


Figure 15, 74: Series 600 Reynolds connector, filled at different pressures with dessicant dried air

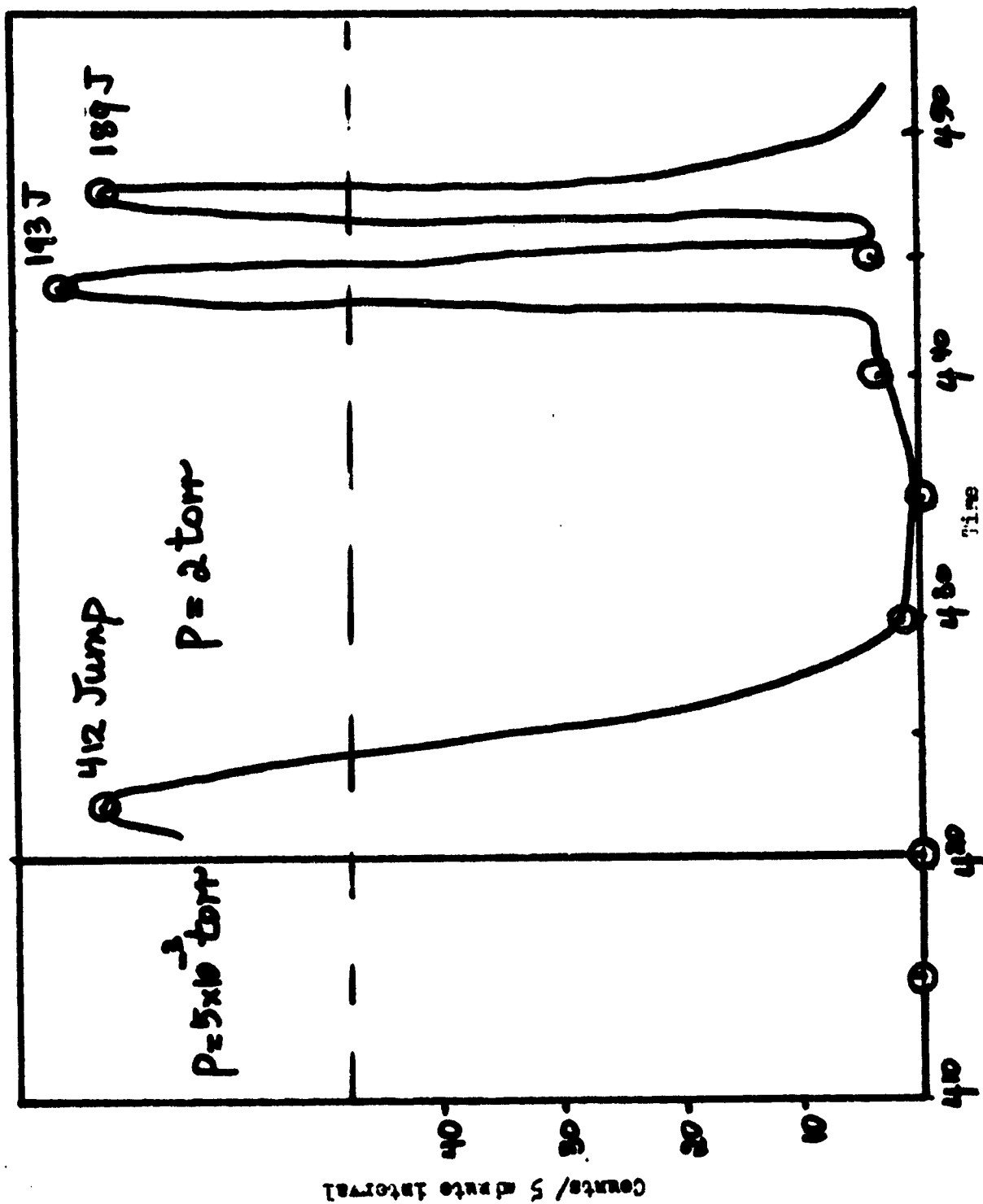


Figure 28a: August 26, 74: Series 600 Reynolds connector after 5 days of continuous outgassing, filled at different pressures with desiccant dried air

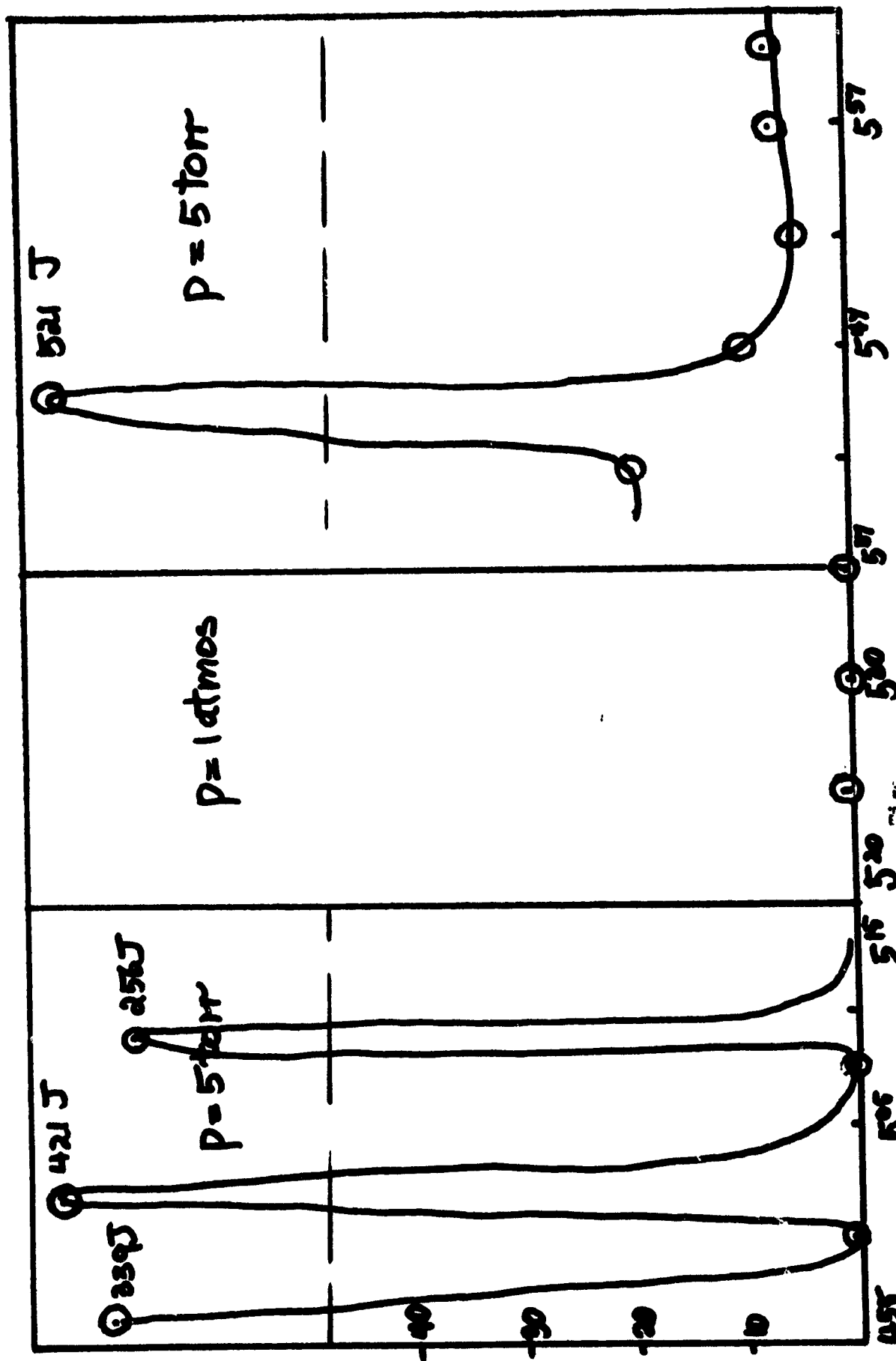
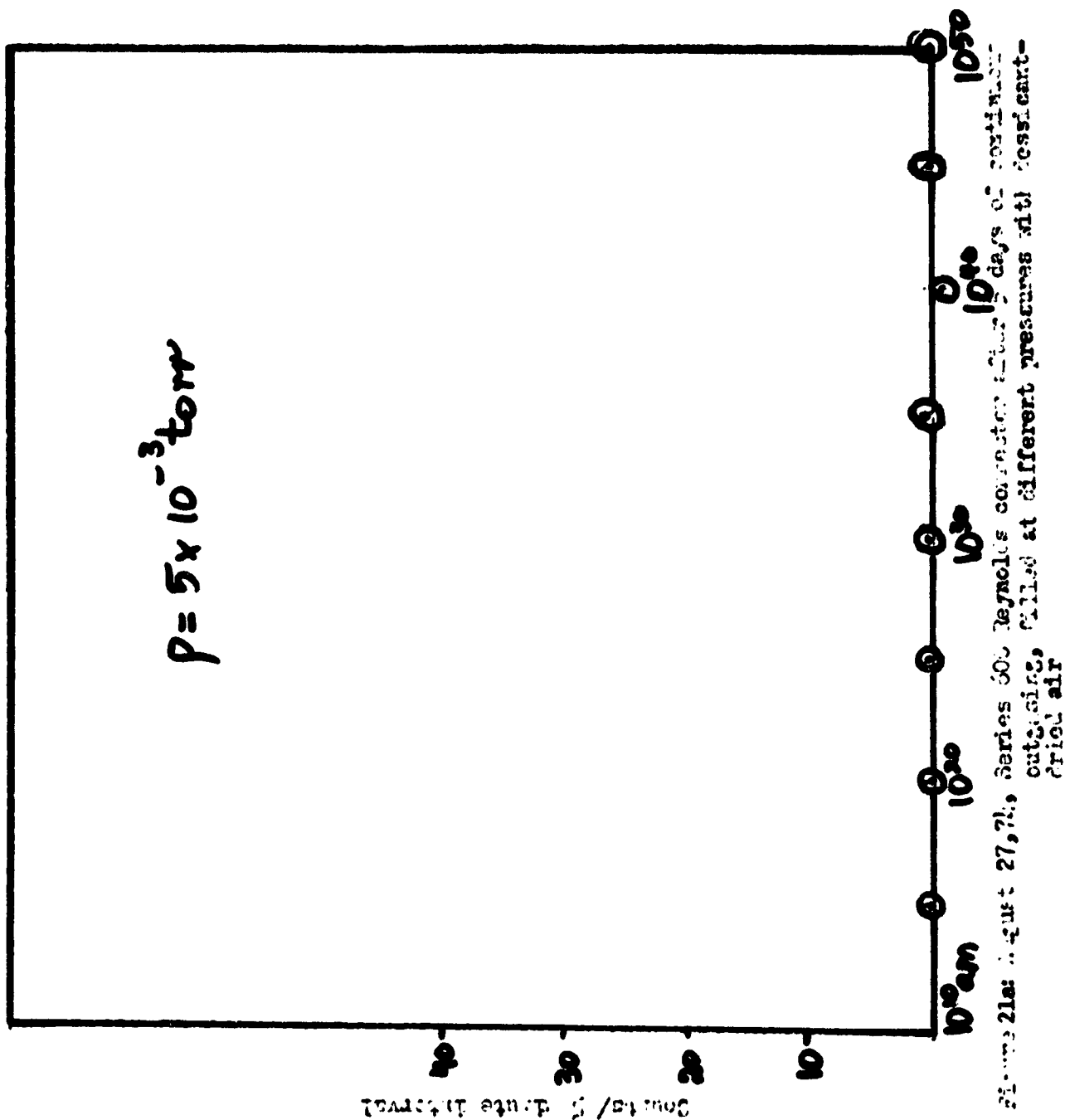


Figure 20: August 26, 74: Ejector 600 cc ejector's corrector after 5 days of continuous outgassing, filled with desiccant-dried air. at different pressures with desiccant-dried air.



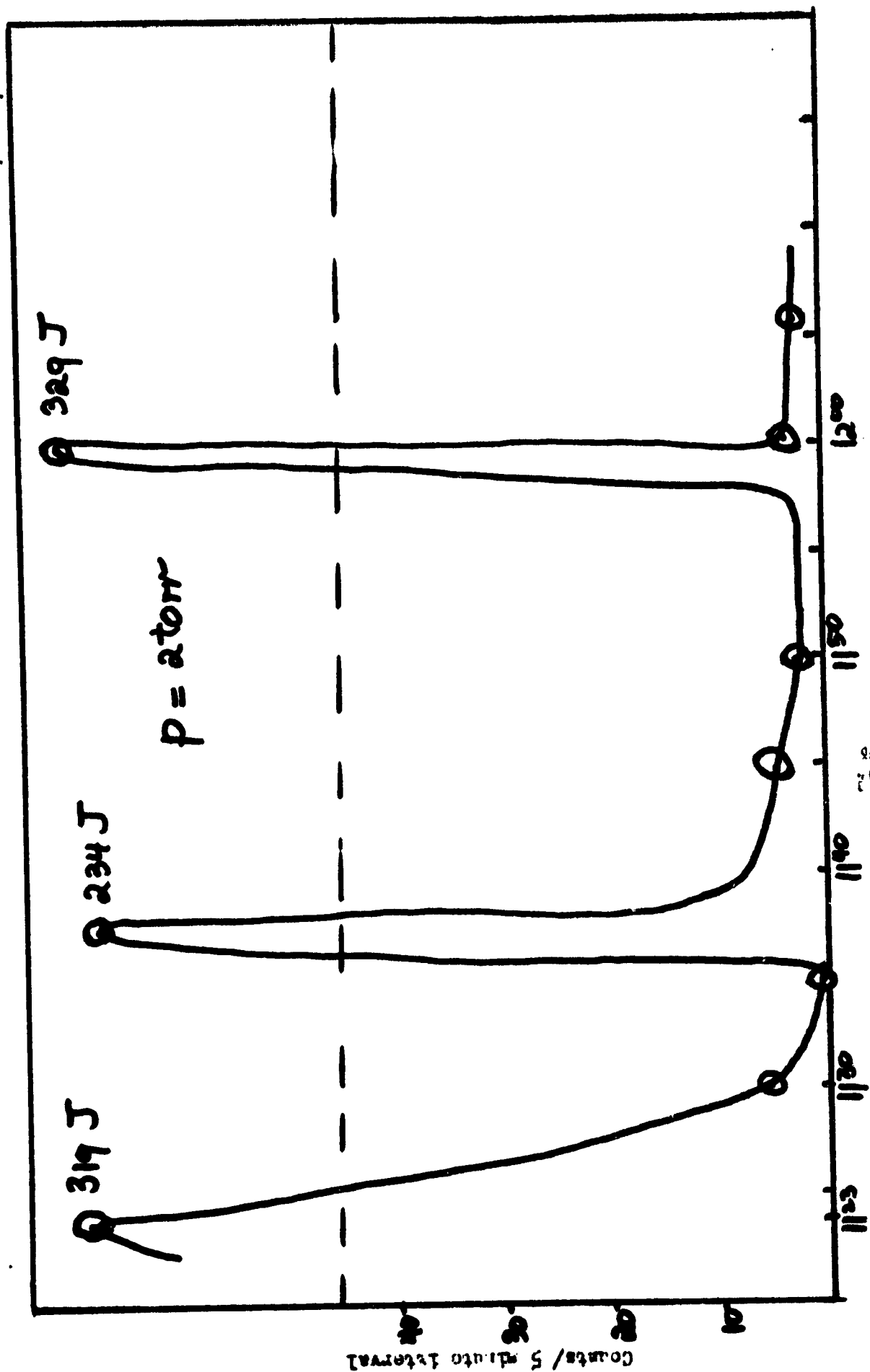


Figure 21b: Experiment 27,74: Series 600 Reynolds 20 micron, after 5 days of continuous outgassing, filled at different pressures with 100% outgassed air

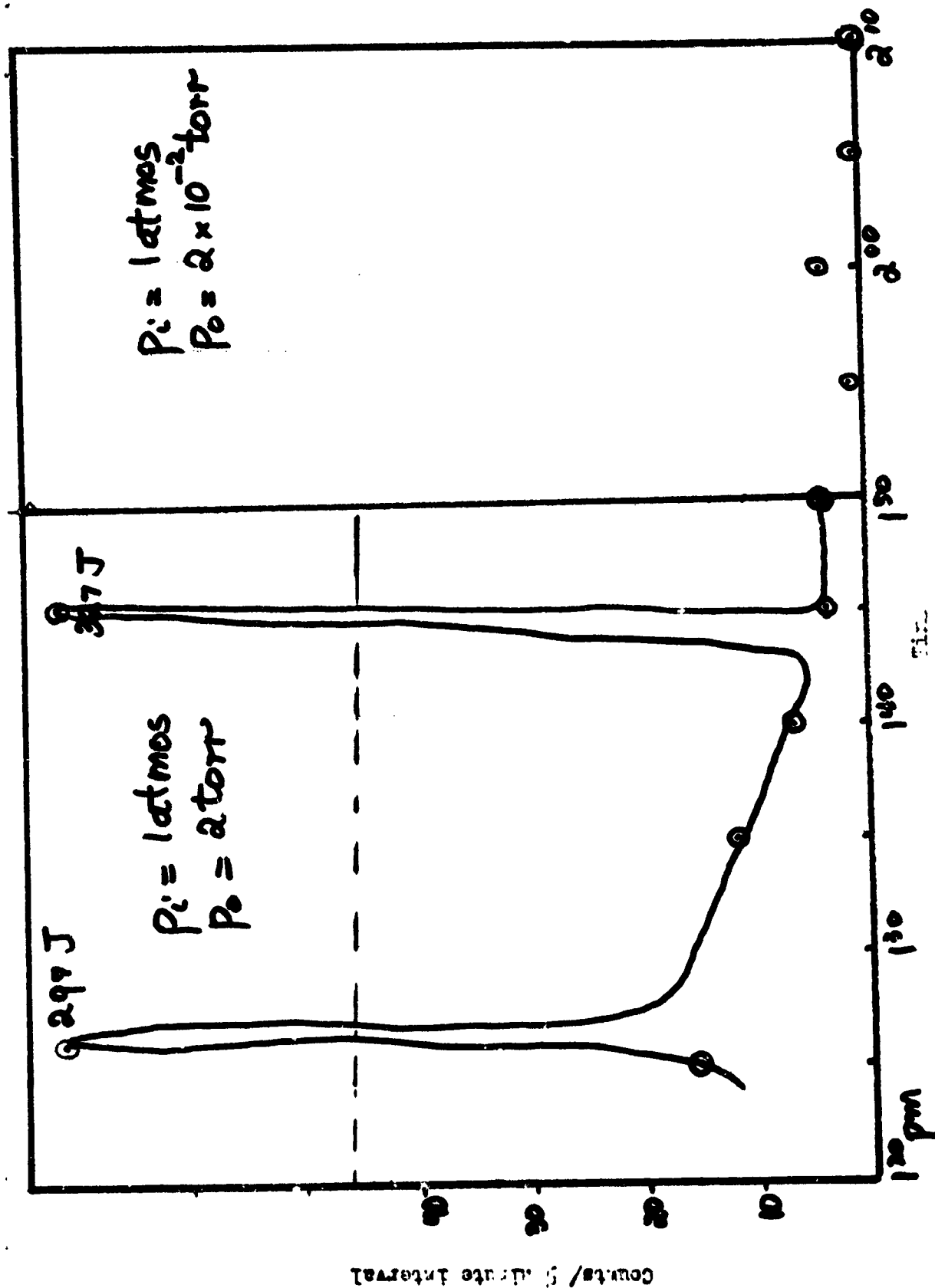


Figure 21c: August 27, 74: Series 600 Reynolds convector, after 5 days of continuous outgassing, filled at different pressures with desiccant-dried air

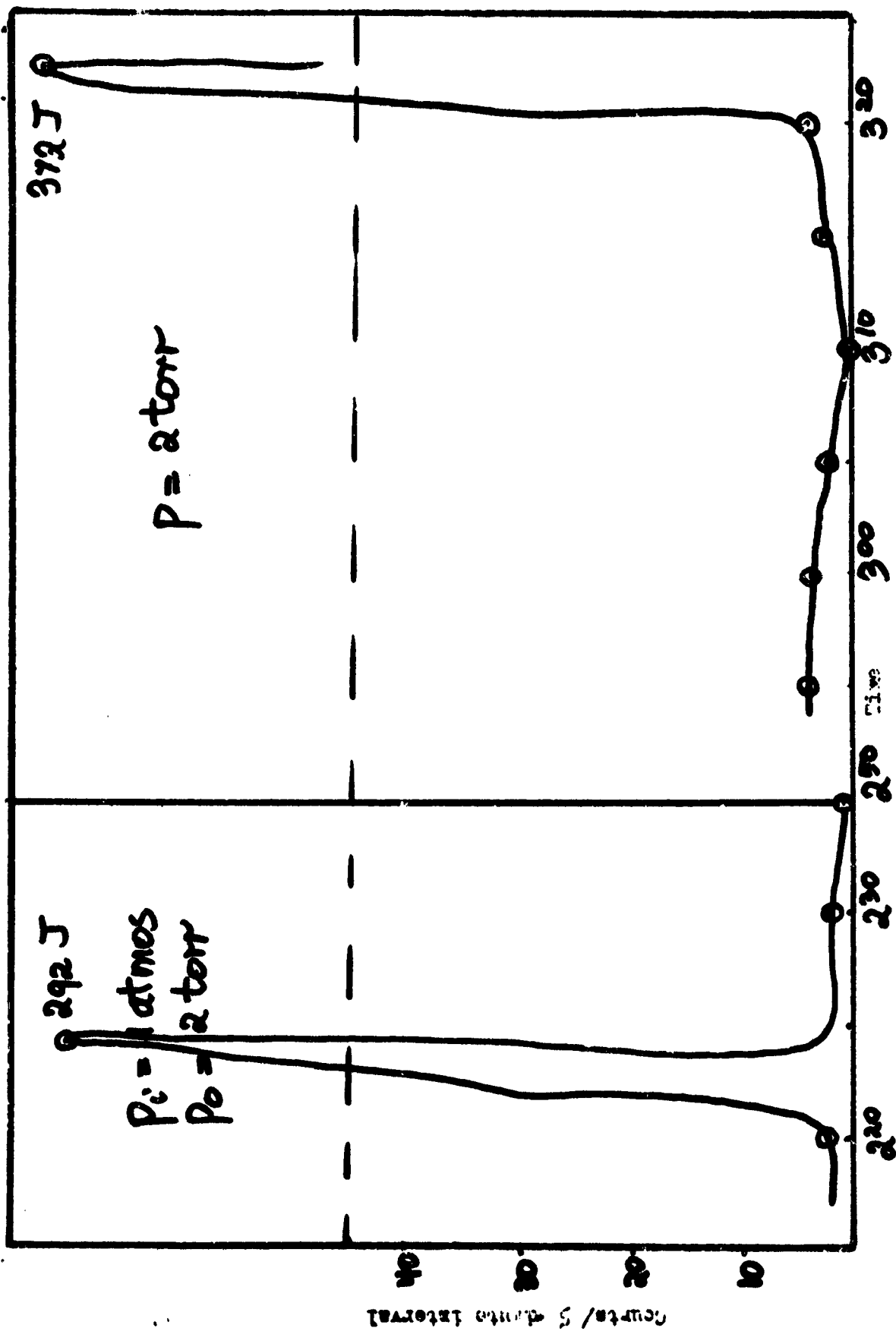


Figure 21d: August 27, 7h: Series 600 Reynolds corrector, after 5 days of continuous outgassing, filled with high-purity pressures with desiccant-dried air

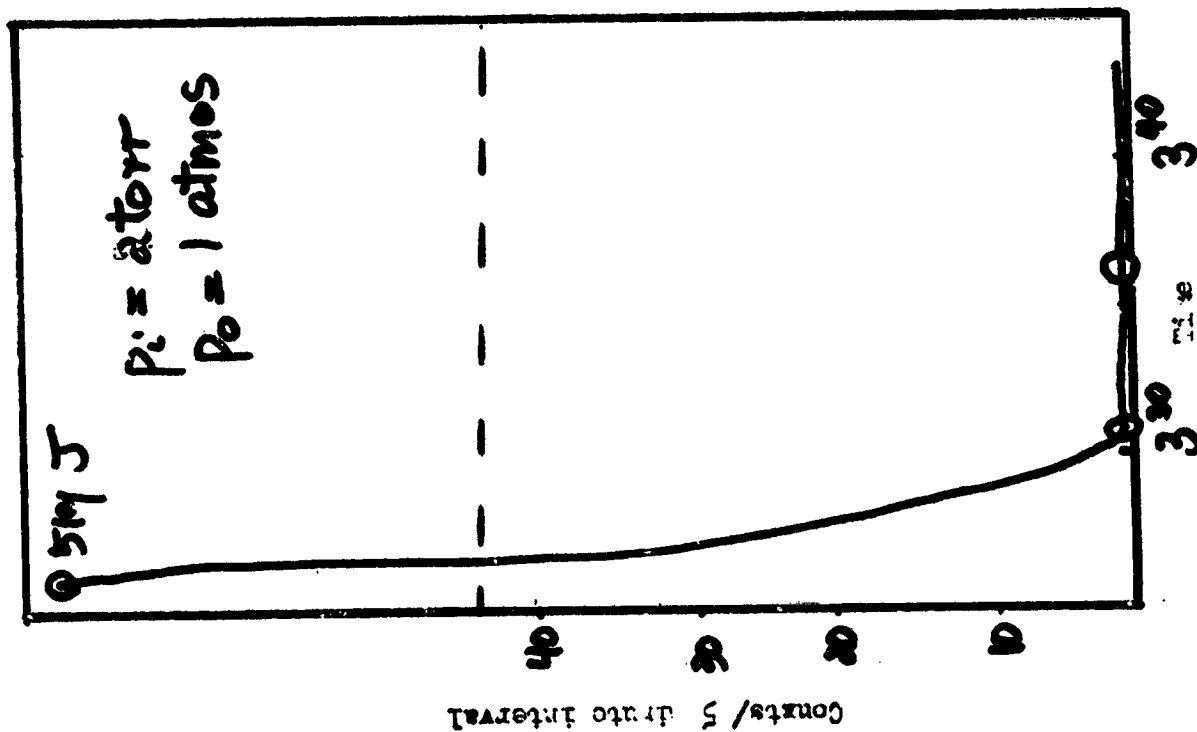


Fig. 21e: August 27, 74: Series 600 Reynolds converter, after 5 days outgassing, filled at different pressures with constant-dried air

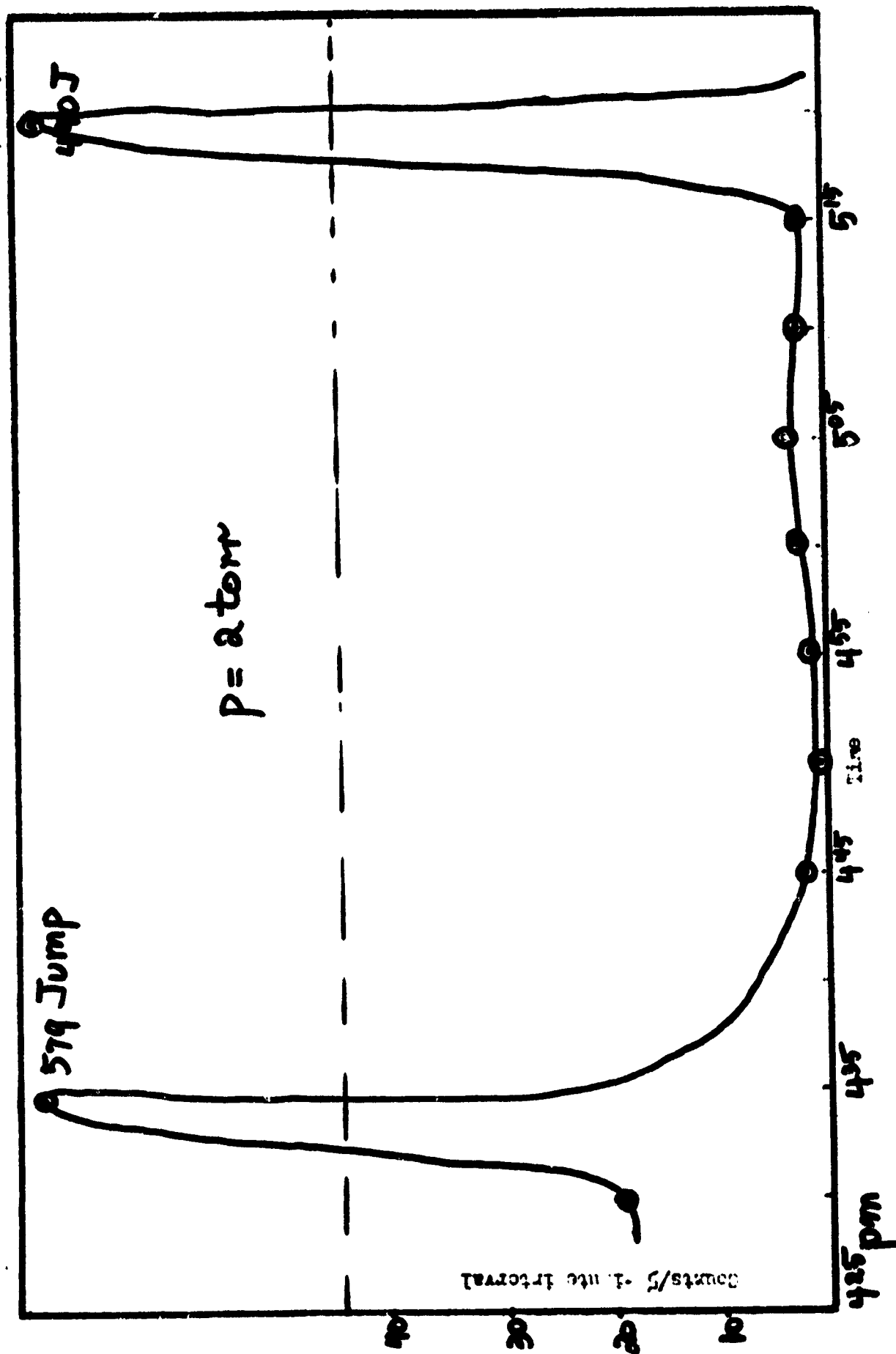


Figure 22 : August 28, 74: Series 500 Reynolds corrector, after 5 days of outgassing, filled at different pressures with desiccant-dried air.

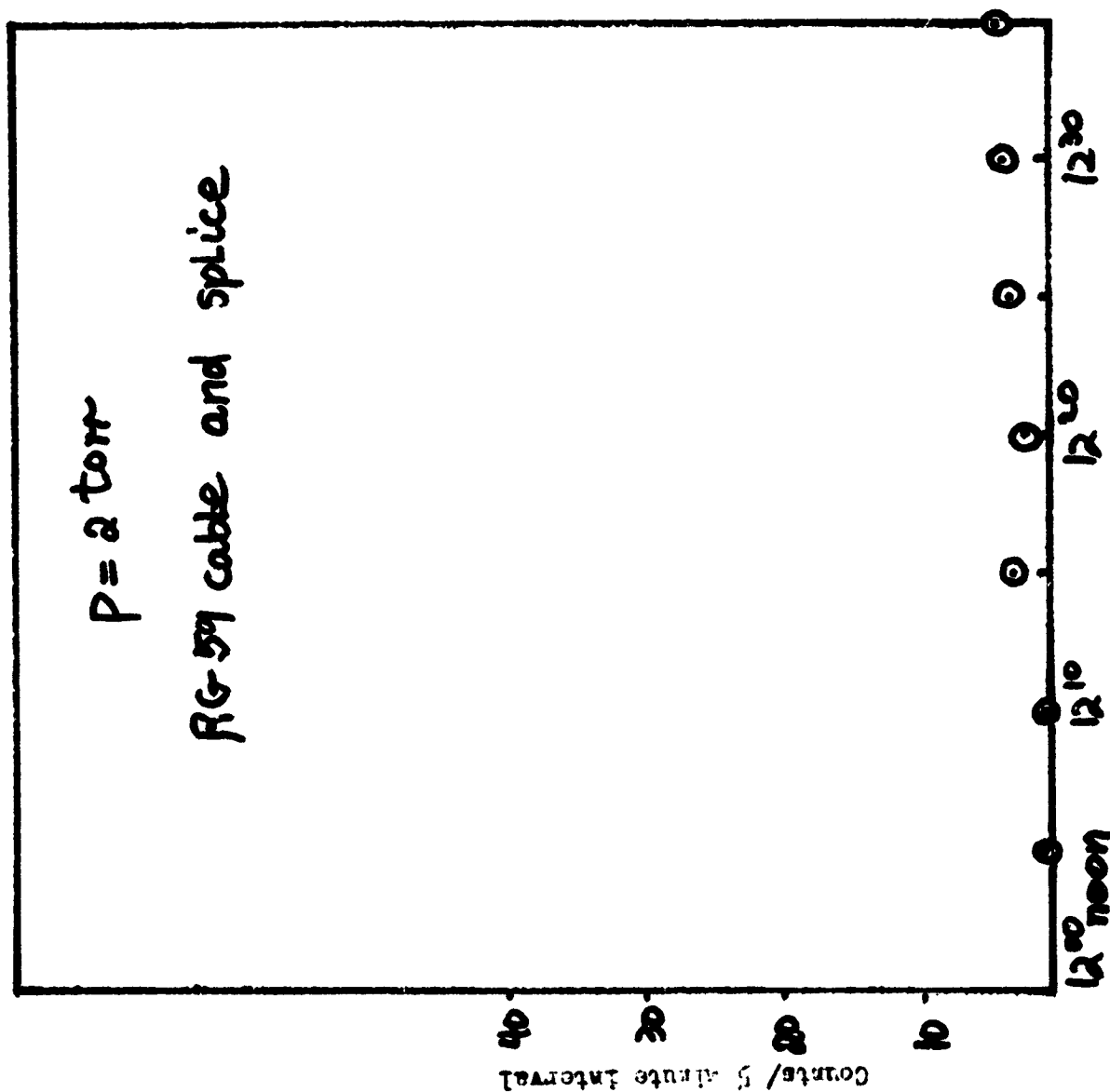


Figure A3: August 29, 74: RG-59 cable and splice at 2 torr

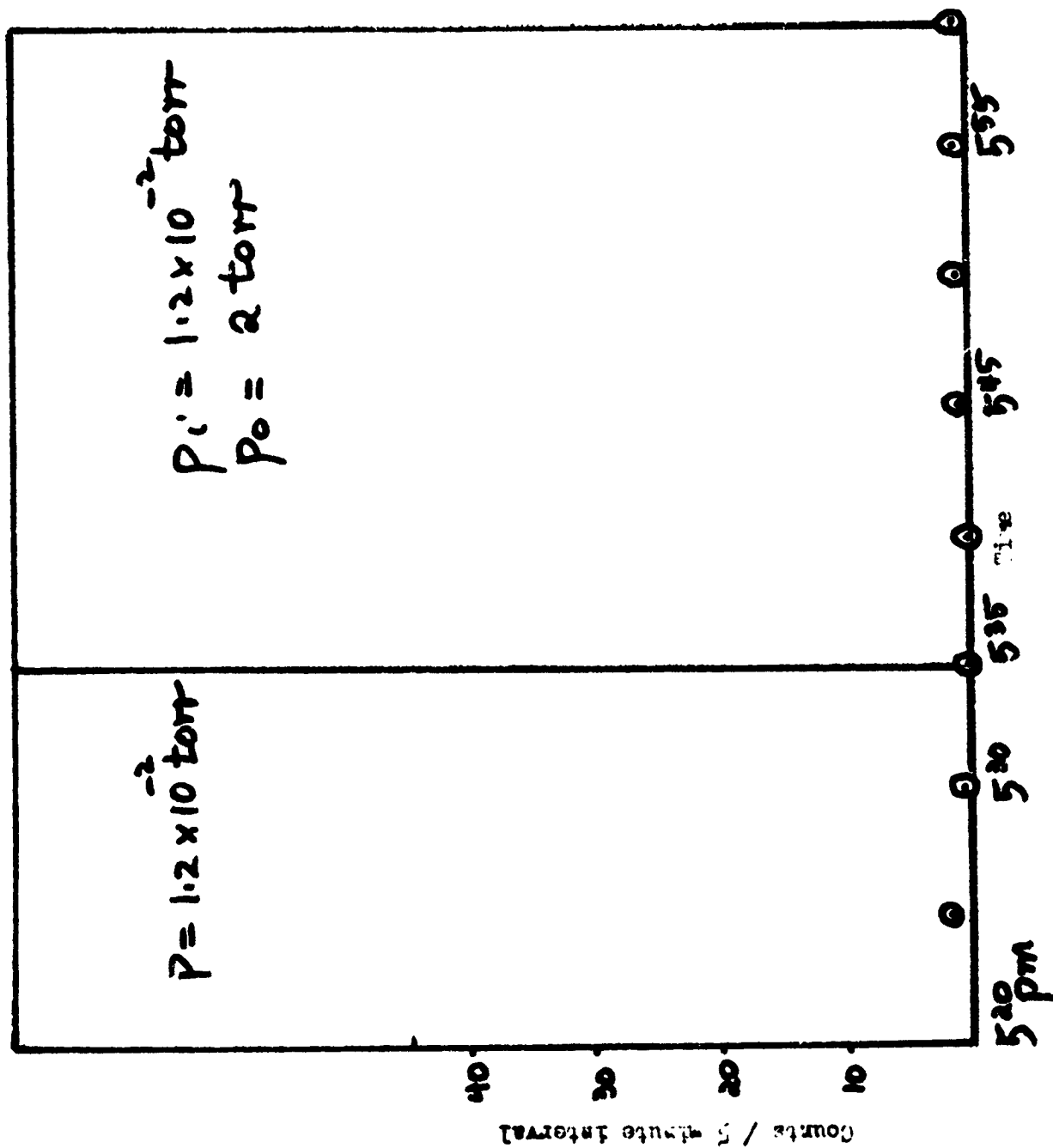


Figure 24a : Series 000. Reynolds connector. Where cable fits into connector it is potted in Stycast 3050 epoxy. Dry nitrogen used as leak test gas. September 16, 74.

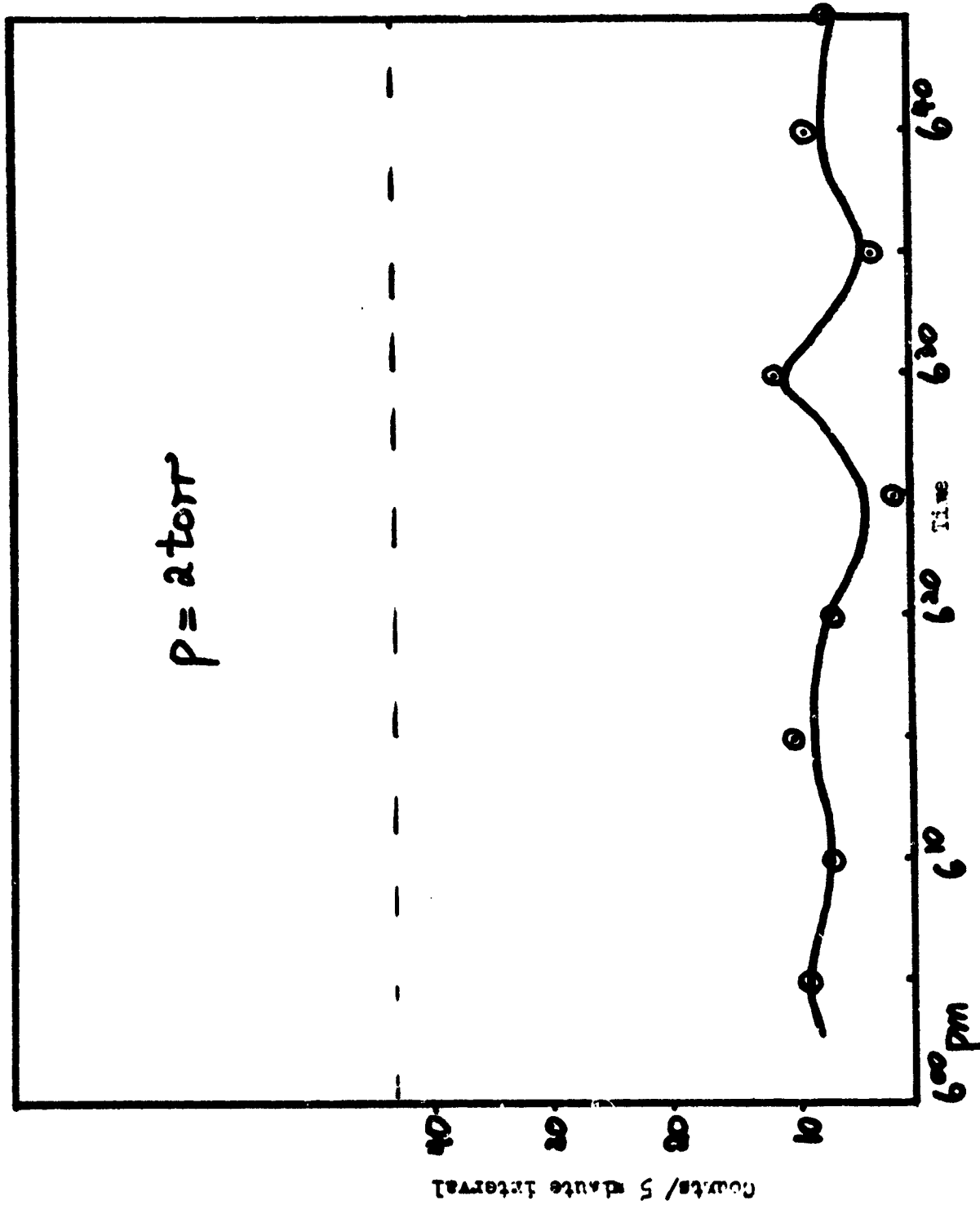


Figure 24b : Series 600 Reynolds corrector. Where cable fits into corrector it is potted in Stycast 3050 epoxy. Dry nitrogen used as leak-in gas. September 16, 74

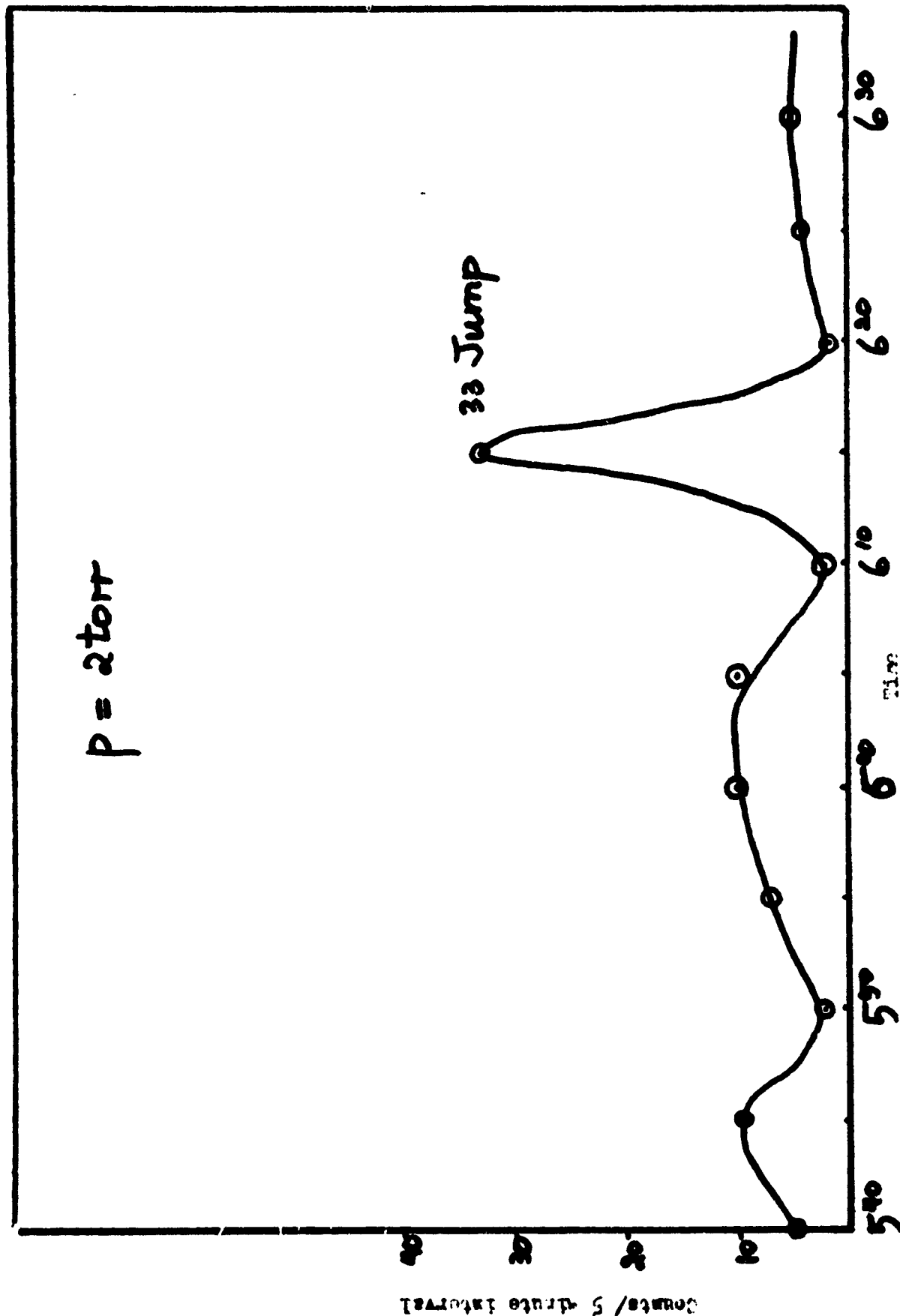


Figure 24c : Coric 600 Reynolds connector. More cable fits into connector it is potted in Styelast 3050 epoxy. Dry nitrogen used as leak-dry gas. September 17, 1971.

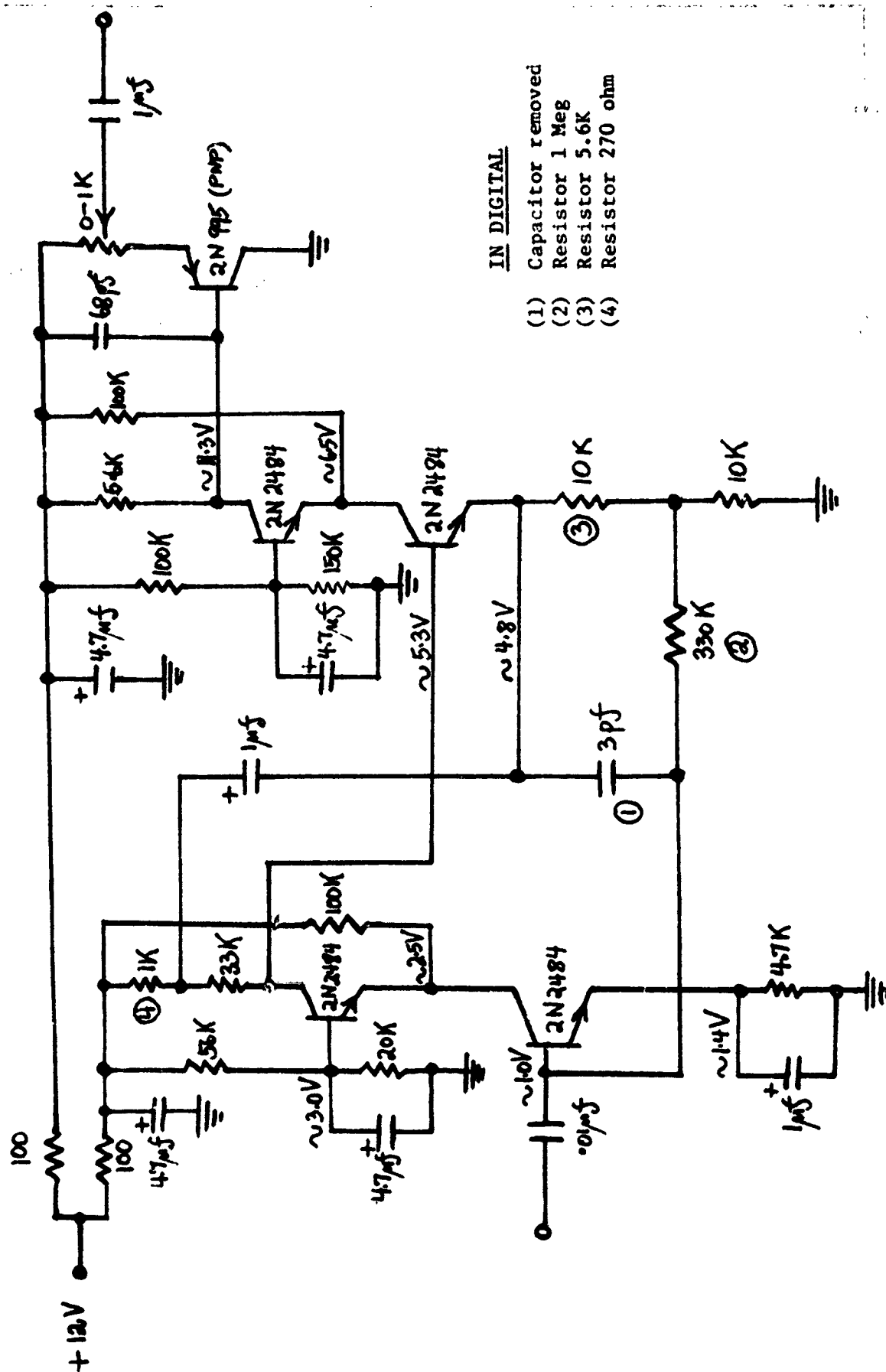


Figure 25: Charge Sensitive Preamplifier
(Courtesy, Mr. Karageorge Mr. Birsa, GSFC)

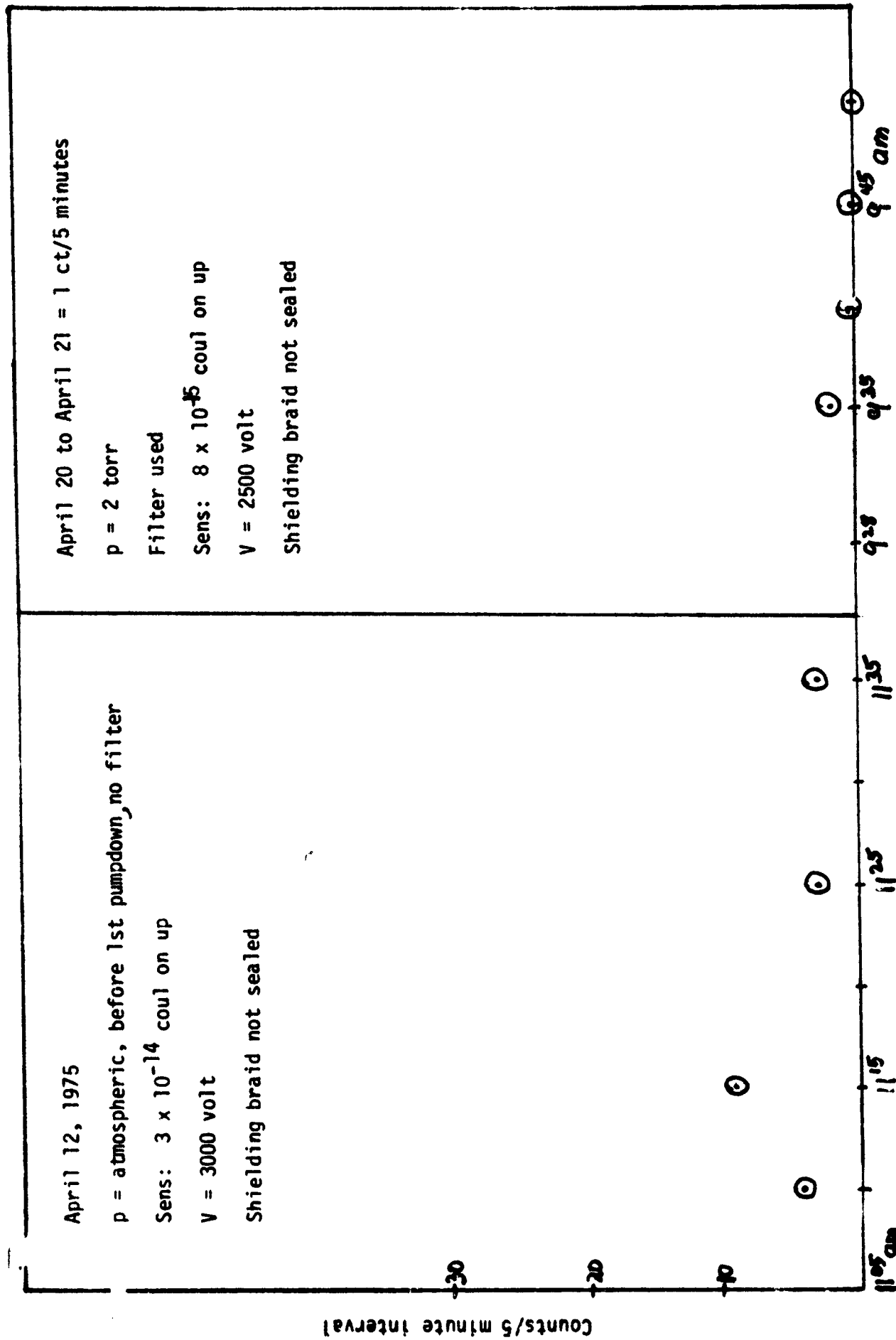


Figure 26: 3 ft. of Reynolds "L" type cable